10/602,929

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NEWS	4	FEB 28	PATDPAFULL - New display fields provide for legal status data from INPADOC
NEWS	5	FEB 28	BABS - Current-awareness alerts (SDIs) available
NEWS	6	FEB 28	MEDLINE/LMEDLINE reloaded
NEWS	7	MAR 02	GBFULL: New full-text patent database on STN
NEWS	8	MAR 03	REGISTRY/ZREGISTRY - Sequence annotations enhanced
NEWS	9	MAR 03	MEDLINE file segment of TOXCENTER reloaded
NEWS	10	MAR 22	KOREAPAT now updated monthly; patent information enhanced
NEWS	11	MAR 22	Original IDE display format returns to REGISTRY/ZREGISTRY
NEWS	12	MAR 22	PATDPASPC - New patent database available
NEWS	13	MAR 22	REGISTRY/ZREGISTRY enhanced with experimental property tags
NEWS	14	APR 04	EPFULL enhanced with additional patent information and new fields
NEWS	15	APR 04	EMBASE - Database reloaded and enhanced

NEWS EXPRESS	JANUARY 10 CURRENT WINDOWS VERSION IS V7.01a, CURRENT MACINTOSH VERSION IS V6.0c(ENG) AND V6.0Jc(JP), AND CURRENT DISCOVER FILE IS DATED 10 JANUARY 2005
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STRUCTURE FILE UPDATES: 10 APR 2005 HIGHEST RN 848184-66-7
DICTIONARY FILE UPDATES: 10 APR 2005 HIGHEST RN 848184-66-7

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* effective March 20, 2005.  A new display format, IDERL, is now     *
* available and contains the CA role and document type information.  *
*                                                                    *
**********************************************************************
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Crossover limits have been increased. See HELP CROSSOVER for details.

Experimental and calculated property data are now available. For more
information enter HELP PROP at an arrow prompt in the file or refer
to the file summary sheet on the web at:
http://www.cas.org/ONLINE/DBSS/registryss.html

```
=> s dibenzylamine/cn
L1            1 DIBENZYLAMINE/CN

=> d l1
```

L1 ANSWER 1 OF 1 REGISTRY COPYRIGHT 2005 ACS on STN
RN 103-49-1 REGISTRY
ED Entered STN: 16 Nov 1984
CN Benzenemethanamine, N-(phenylmethyl)- (9CI) (CA INDEX NAME)
OTHER CA INDEX NAMES:
CN Dibenzylamine (8CI)
OTHER NAMES:
CN (N-Benzylaminomethyl)benzene
CN Bibenzylamine
CN DBA
CN N,N-Dibenzylamine
CN N-(Phenylmethyl)benzenemethanamine
CN N-Benzylbenzylamine
CN NSC 4811
FS 3D CONCORD
DR 306991-23-1
MF C14 H15 N
CI COM
LC STN Files: AGRICOLA, ANABSTR, AQUIRE, BEILSTEIN*, BIOBUSINESS, BIOSIS,
BIOTECHNO, CA, CANCERLIT, CAOLD, CAPLUS, CASREACT, CBNB, CEN, CHEMCATS,
CHEMINFORMRX, CHEMLIST, CSCHEM, DDFU, DETHERM*, DRUGU, EMBASE, GMELIN*,
HODOC*, IFICDB, IFIPAT, IFIUDB, IPA, MEDLINE, MRCK*, NIOSHTIC, PIRA, PS,
SPECINFO, SYNTHLINE, TOXCENTER, USPAT2, USPATFULL
(*File contains numerically searchable property data)
Other Sources: DSL**, EINECS**, TSCA**
(**Enter CHEMLIST File for up-to-date regulatory information)

PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT

2022 REFERENCES IN FILE CA (1907 TO DATE)
49 REFERENCES TO NON-SPECIFIC DERIVATIVES IN FILE CA
2031 REFERENCES IN FILE CAPLUS (1907 TO DATE)
16 REFERENCES IN FILE CAOLD (PRIOR TO 1967)

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                                                           ENTRY    SESSION
FULL ESTIMATED COST                                         6.87       7.08

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FILE COVERS 1907 - 11 Apr 2005  VOL 142 ISS 16
FILE LAST UPDATED: 10 Apr 2005  (20050410/ED)

  This file contains CAS Registry Numbers for easy and accurate
  substance identification.


=> s 103-49-1/rn
          2031 103-49-1
            49 103-49-1D
L2        1990 103-49-1/RN
                (103-49-1 (NOTL) 103-49-1D )

=> s ?color
L3      408778 ?COLOR

=> s ?colour
L4        1791 ?COLOUR

=> s l3 or l4
L5      409531 L3 OR L4

=> s l2 and l5
L6          28 L2 AND L5

=> d l6 1-28 abs ibib
```

L6 ANSWER 1 OF 28 CAPLUS COPYRIGHT 2005 ACS on STN

AB Dibenzylamine having a color value of ≤100 Hazen units is manufactured by the addition of ammonium chloride or amines to the pre-distilled reaction mixture followed by distillation

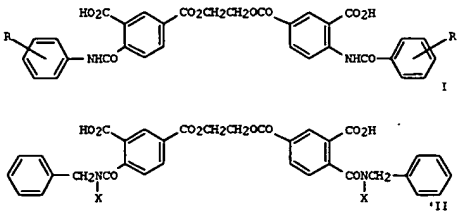
ACCESSION NUMBER: 2004:5167 CAPLUS
DOCUMENT NUMBER: 140:78820
TITLE: Process for the preparation of colorless dibenzylamine
INVENTOR(S): Heuer, Lutz
PATENT ASSIGNEE(S): Bayer Chemicals Ag, Germany
SOURCE: Eur. Pat. Appl., 4 pp.
CODEN: EPXXDW
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 1375470	A2	20040102	EP 2003-13535	20030613
EP 1375470	A3	20040929		
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, HU, SK				
DE 10228594	A1	20040122	DE 2002-10228594	20020626
JP 2004026830	A2	20040129	JP 2003-179580	20030624
US 2004026226	A1	20040212	US 2003-602929	20030624
CN 1470495	A	20040128	CN 2003-145230	20030625

PRIORITY APPLN. INFO.: DE 2002-10228594 A 20020626
OTHER SOURCE(S): MARPAT 140:78820

L6 ANSWER 2 OF 28 CAPLUS COPYRIGHT 2005 ACS on STN

GI



I

II

AB The material comprises a support having a heat-sensitive layer containing a leuco dye and I or II (R = H, COOH; X = H, benzyl) as a color developer. The material shows good storage stability and gives images with oil resistance.

ACCESSION NUMBER: 1999:406902 CAPLUS
DOCUMENT NUMBER: 131:80809
TITLE: Thermal printing material containing leuco dye and benzamide derivative color developer
INVENTOR(S): Morita, Mitsunobu; Hayakawa, Kunio
PATENT ASSIGNEE(S): Ricoh Co., Ltd., Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 10 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 11170708	A2	19990629	JP 1997-364067	19971217

PRIORITY APPLN. INFO.: JP 1997-364067 19971217
OTHER SOURCE(S): MARPAT 131:80809

L6 ANSWER 3 OF 28 CAPLUS COPYRIGHT 2005 ACS on STN

AB Photochem. Al film dissoln. is studied in polymeric electron donor-acceptor layers containing thiadicarbocyanine dye sensitizer either in a monomeric or in J-aggregate form. Novolak resin or poly(vinylethylal) were used as polymer matrixes, dibenzylamine and ferrocene as electron donors, and CBr4 as electron acceptor. Quantum yield of the sensitized color product formation in polymer layer was higher in the layers containing dye aggregates. Dissoln. of Al in polymer layer was only observed in the presence of dye J-aggregates.

ACCESSION NUMBER: 1998:237507 CAPLUS
DOCUMENT NUMBER: 128:328689
TITLE: Role of dye J-aggregates in photochemical dissolution of aluminum in polymer donor-acceptor layers
AUTHOR(S): Grishina, A. D.; Pereshivko, L. Ya.; Tedoradze, M. G.; Shapiro, B. I.
CORPORATE SOURCE: Inst. Elektrokhim. im. Frumkina, RAN, Moscow, Russia
SOURCE: Zhurnal Nauchnoi i Prikladnoi Fotografii (1998), 43(2), 19-25
CODEN: ZNPFEK; ISSN: 0869-6144
PUBLISHER: Nauka
DOCUMENT TYPE: Journal
LANGUAGE: Russian

L6 ANSWER 4 OF 28 CAPLUS COPYRIGHT 2005 ACS on STN

GI

AB The photog. material contains a binder, a photog. Ag halide, and (A) RNH2 and/or R1NHR2 (R = C≥6 aliphatic group, C≥6 aromatic group, C≥6 heterocycle, C≥6 polymer chain; R1, R2 = aliphatic group, aromatic group, polymer chain; total C number in R1 and R2 ≥6; R1 and R2 may form a ring) or (B) R3CH2R4 (R3, R4 = acyl, carbamoyl, alkoxycarbonyl, aryloxycarbonyl, NO2, cyano, SO3H, Q; total C number in R3 and R4 ≥6; R3 and R4 may form a ring; Z = atomic group to form a N-containing heterocycle) and a dye-donating substance which releases a diffusible dye by reaction with Ag+ or a soluble Ag+ complex under an alkali condition on a support. The photog. material showed improved whiteness of the base color and good storage stability.

ACCESSION NUMBER: 1997:171837 CAPLUS
DOCUMENT NUMBER: 126:178979
TITLE: Diffusion-transfer heat-developable color photographic material containing primary or secondary amine
INVENTOR(S): Ushiku, Masayuki; Myazawa, Kazuhiro; Ooya, Hidenobu; Oohayashi, Keiji
PATENT ASSIGNEE(S): Konishiroku Photo Ind, Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 32 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 08334879	A2	19961217	JP 1995-137943	19950605

PRIORITY APPLN. INFO.: JP 1995-137943 19950605

L6 ANSWER 5 OF 28 CAPLUS COPYRIGHT 2005 ACS on STN
GI For diagram(s), see printed CA Issue.
AB Photothermog. elements incorporate leuco forms of phenazinium dyes to provide a developed color image. The dye has the general formula I [R1, R2, R4, R11 = H, R12, SO2R12, COR12, or NR1R2 = heterocyclyl; R3, R5-R9 = H, R12, heterocyclyl, CN, OH, OR12, halo, NO2, SH, SR12, SO2R12, COR12, acyloxy, SO2NH2, or combinations represent fused (hetero)aromatic rings containing C, N, O, and/or S; R10 is any group which will not prevent oxidative cleavage of the X1-N bond; R12 = alkyl, aryl; X1 = CO, CONR11, CO2, SO2; X2 = H, any substituent other than (substituted) amino; when R1 is Et, R2 is not C2H4NHSO2Me]. Thus, phenazine was quaternized with Et2SO4, oxidized with K3Fe(CN)6 in aqueous NaOH, chlorinated with POCl3/PCl5, and condensed with PhCHZNHMe to give I (R1 = PhCH2, R2 = Me, R3 = R5-R9 = X2 = H, R4 = Et, R10 = Ph, X1 = CO), a leuco dye which can be developed to a magenta shade.
ACCESSION NUMBER: 1995:994399 CAPLUS
DOCUMENT NUMBER: 124:32011
TITLE: Monoaminophenazine leuco dyes and photothermographic materials containing them
INVENTOR(S): Grieve, Duncan; Mott, Andrew W.; Nairne, Robert J. D.; Bays, David C.; Poon, Stephen S. C.; Attwood, Martin D.; Jackson, Andrew C.
PATENT ASSIGNEE(S): Minnesota Mining and Manufacturing Co., USA
SOURCE: Eur. Pat. Appl., 27 pp.
CODEN: EPXXDW
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 671393	A1	19950913	EP 1995-301483	19950307
R: DE, FR, GB, IT				
JP 07258561	A2	19951009	JP 1995-51052	19950310
PRIORITY APPLN. INFO.:			GB 1994-4806 A	19940311
OTHER SOURCE(S):	MARPAT 124:32011			

L6 ANSWER 6 OF 28 CAPLUS COPYRIGHT 2005 ACS on STN
AB A powdered flame retardant, which does not impair the transparency or phys. properties of the title resins, comprises 5-30 parts alkali metal (Li, Na, K) compound, 0.2-10 parts perchloric acid radical in the form of the acid, salt or amine thereof, and 1-50 parts hydrophobic dispersant (b. ≥200°) based on 100 parts Sb2O5. A PVC composition containing 7 phr flame retardant of Sb2O5 100, Na2O 14.4, perchloric acid as ClO4 3.5, polyoxyethylene dodecylamine (I) 8.0, and H2O 16.4% was formed into a test specimen having thermal stability (darkening time at 185°) 180 min and initial color (YI value) 8.9, vs. 135 and 13.4, resp., for flame retardant containing Sb2O5 100, Na2O 15.2, ClO4 3.6, and I 0.4 parts.
ACCESSION NUMBER: 1993:582145 CAPLUS
DOCUMENT NUMBER: 119:182145
TITLE: Flame retardant for halogen-containing vinyl resins
INVENTOR(S): Watanabe, Yoshitane; Suzuki, Keitaro; Shishido, Kouji; Teranishi, Masayuki; Shindo, Masuo
PATENT ASSIGNEE(S): Nissan Chemical Industries, Ltd., Japan
SOURCE: U.S., 13 pp. Cont.-in-part of U.S. Ser. No. 311,524, abandoned.
CODEN: USXXAM
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 2
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 5190700	A	19930302	US 1990-574606	19900829
PRIORITY APPLN. INFO.:			JP 1988-42640 A	19880225
			US 1989-311524 B2	19890216

L6 ANSWER 7 OF 28 CAPLUS COPYRIGHT 2005 ACS on STN
AB IR spectra are analyzed of the products of photoinduced electron-transfer in the donor-acceptor complexes of aromatic amines (diphenylamine, dibenzylamine) with hexabromodimethyl sulfone. The color image forming products were diphenylmethane dye (in the case of diphenylamine-containing system), and N,N-diphenylphenylmethyleneimine bromide (dibenzylamine system).
ACCESSION NUMBER: 1992:500747 CAPLUS
DOCUMENT NUMBER: 117:100747
TITLE: IR spectra of the photodissociation products of complexes from charge transfer between aromatic amines and bromine-containing acceptors
AUTHOR(S): Grishina, A. D.; Tedoradze, M. G.; Vannikov, A. V.
CORPORATE SOURCE: Inst. Elektrokhim. im. Frumkina, Moscow, Russia
SOURCE: Zhurnal Nauchnoi i Prikladnoi Fotografii (1992), 37(1), 54-61
CODEN: ZNPFEK; ISSN: 0869-6144
DOCUMENT TYPE: Journal
LANGUAGE: Russian

L6 ANSWER 8 OF 28 CAPLUS COPYRIGHT 2005 ACS on STN
AB Title polymer, useful for blending with styrene resins, has improved flow, color and odor and is prepared by oxidizing 2,6-dialkyl-4-halophenol in presence of a H2O-immiscible solvent, aqueous alkali, phase-transfer agent, and an amine containing ≥1 H on a N and directly bonded by aliphatic C atom(s) (mol.-weight control agent). Thus, oxidative polymerization of 4-bromo-2,6-dimethylphenol, 6 M NaOH in PhMe in presence of Bu4NHSO4 and Bu2NH in air at room temperature, neutralizing with AcOH, and adding the organic phase to MeOH precipitated polymer with intrinsic viscosity (CHCl3, 25°) 0.40 dL/g and 0.065% N.
ACCESSION NUMBER: 1992:175405 CAPLUS
DOCUMENT NUMBER: 116:175405
TITLE: Polyphenylene ether process and resin composition
INVENTOR(S): Shaffer, Timothy D.; Bennett, James G., Jr.; Denniston, Mark R.
PATENT ASSIGNEE(S): General Electric Co., USA
SOURCE: U.S., 8 pp.
CODEN: USXXAM
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 5084551	A	19920128	US 1990-626598	19901212
EP 490164	A2	19920617	EP 1991-120143	19911126
EP 490164	A3	19930616		
R: DE, ES, FR, GB, IT, NL				
JP 05009290	A2	19930119	JP 1991-349457	19911209
JP 07051624	B4	19950605		
PRIORITY APPLN. INFO.:			US 1990-626598 A	19901212

L6 ANSWER 9 OF 28 CAPLUS COPYRIGHT 2005 ACS on STN

AB Polyoxyphenylenes with good color and, in blends with high-impact polystyrene, good impact strength are prepared by oxidative polymerization of the phenols 2-R1-3-R3-6-R2C6H2OH [R1 = C1-4 hydrocarbyl, (substituted) Ph; R2 = the groups of R1 or halogen; R3 = the groups of R2 or H] in the presence of MeOH or EtOH, Cu compds., tetramethylpropanediamine derivs., and Br or Cl compds. Passing O into a mixture of 2.05 mg Cu2O, 27.5 mg 35% HCl, 12.6 g MeOH, 0.1495 g N,N,N',N'-tetramethyl-1,3-propanediamine, 7.0 g 2,6-xylenol, 37.8 g PhMe, and 12.6 g BuOH stirred at 30° for 3.5 h gave a polyoxyphenylene with reduced sp. viscosity 0.53.

ACCESSION NUMBER: 1988:550250 CAPLUS
DOCUMENT NUMBER: 109:150250
TITLE: Polymerization catalysts for the preparation of polyoxyphenylenes
INVENTOR(S): Ibe, Sadao; Sakurai, Tokio; Takahashi, Kazuhiro; Unno, Yoshiro
PATENT ASSIGNEE(S): Asahi Chemical Industry Co., Ltd., Japan
SOURCE: Ger. Offen., 19 pp.
CODEN: GWXXBX
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 2
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 3741038	A1	19880609	DE 1987-3741038	19871203
DE 3741038	C2	19900308		
JP 63142029	A2	19880614	JP 1986-287788	19861204
US 4788277	A	19881129	US 1987-127842	19871202
NL 8702910	A	19880701	NL 1987-2910	19871203
NL 188097	B	19911101		
NL 188097	C	19920401		
CN 87107289	A	19880615	CN 1987-107289	19871204
CN 1008101	B	19900523		
JP 01158035	A2	19890621	JP 1988-28684	19880212
JP 05013964	B4	19930223		

PRIORITY APPLN. INFO.:

JP 1986-287788	A	19861204
JP 1987-29591	A	19870213
JP 1987-77570	A	19870401
JP 1987-216449		19870901

L6 ANSWER 10 OF 28 CAPLUS COPYRIGHT 2005 ACS on STN

AB Time-resolved spectral changes were studied in flash (20 μs) UV photolysis of the films and dichloromethane solns. containing poly(vinyl alc.), an aromatic amine (dibenzylamine, triphenylamine, diphenylbenzylamine) and CBr2. The stable colored photoproducts (absorption maximum .apprx.650 nm) were absent in the 1st 160 μs after the photolyzing pulse. These products were formed in the later secondary reaction steps in these systems.

ACCESSION NUMBER: 1987:449270 CAPLUS
DOCUMENT NUMBER: 107:49270
TITLE: Study of the early stages of the mechanism of formation of color in the presence of light in polymeric films containing aromatic amines and carbon tetrabromide
AUTHOR(S): Mal'tsev, E. I.; Kolotilkin, A. S.; Kruglov, A. B.
CORPORATE SOURCE: Inst. Elektrokhim., Moscow, USSR
SOURCE: Elektron. Org. Mater. (1985), 316-18
CODEN: 55TIAF
DOCUMENT TYPE: Conference
LANGUAGE: Russian

L6 ANSWER 11 OF 28 CAPLUS COPYRIGHT 2005 ACS on STN

AB A liquid developer for diazo copying paper is obtained by dispersing a liquid organic amine (boiling ≥150°) in a silicone oil. The developer gives high image d. and exhibits no adverse effects from temperature and humidity. Thus, octylamine and a silicone oil (KF-96-100; from Shin-Etsu Chemical Co., Ltd.) were mixed to give a diazo copying paper developer (viscosity 30 cP at 20°). An image prepared by using the developer showed a high optical d. of 1.21, and the image did not discolor after extended light exposure.

ACCESSION NUMBER: 1985:70298 CAPLUS
DOCUMENT NUMBER: 102:70298
TITLE: Liquid developer for diazo copying paper
PATENT ASSIGNEE(S): Ricoh Co., Ltd., Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 3 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 59062851	A2	19840410	JP 1982-174839	19821004

PRIORITY APPLN. INFO.:

JP 1982-174839		19821004

L6 ANSWER 12 OF 28 CAPLUS COPYRIGHT 2005 ACS on STN

AB Chelate-type copying materials having outstanding whiteness contain a relatively inexpensive organophosphorus-Fe compound with PO- or PS- bonds with the Fe3+ and a reactive ligand. In the materials, the Fe compound may have a colorless or slightly colored oil-soluble and/or thermally fusible organic compound adhering to its surface or may contain an organic base therein that is not in contact with Fe compound Thus, to a stirred solution containing 4-tert-butylbenzoic acid 89, PhZHPO4 125, Na laurylbenzenesulfonate 70, and 5% aqueous NaOH 800 parts was added an aqueous solution of FeCl3.6H2O 108 in water 500 parts. This dispersion was then mixed with 20% aqueous Na tert-butylbenzoate 500 parts and then TiCl4 25 parts to give a dispersion containing light yellow particles. A coating composition containing these particles 20, Na polyacrylate 1, hydroxyethyl cellulose 1, TiO2 20, CaCO3 60, a carboxylated butadiene-styrene copolymer 15, and water 200 parts was then coated on a paper support at 5 g/m2 to give a copying paper undersheet with a whiteness of 81%. When combined with a copying paper oversheet containing ligand-containing microcapsules, a color image with color d. of 0.95 was obtained.

ACCESSION NUMBER: 1984:601650 CAPLUS
DOCUMENT NUMBER: 101:201650
TITLE: Recording material containing iron salts
INVENTOR(S): Shioi, Shunshuke; Matoba, Gensuke; Miyake, Makoto
PATENT ASSIGNEE(S): Kanzaki Paper Mfg. Co., Ltd., Japan
SOURCE: Ger. Offen., 97 pp.
CODEN: GWXXBX
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 3330679	A1	19840301	DE 1983-3330679	19830825
JP 59038088	A2	19840301	JP 1982-148428	19820825
JP 01005836	B4	19890201		
JP 59038089	A2	19840301	JP 1982-149414	19820828
JP 01003675	B4	19890123		
JP 59064386	A2	19840412	JP 1982-167012	19820925
JP 01003674	B4	19890123		
US 4602264	A	19860722	US 1983-522315	19830811
GB 2130614	A1	19840606	GB 1983-22032	19830816
GB 2130614	B2	19860115		

PRIORITY APPLN. INFO.:

JP 1982-148428	A	19820825
JP 1982-149414	A	19820828
JP 1982-167012	A	19820925

L6 ANSWER 13 OF 28 CAPLUS COPYRIGHT 2005 ACS on STN
AB Thirteen procedures are described for the colorimetric and fluorometric determination of amines. In the presence of an appropriate base, 1,3,5-trinitrobenzene condenses with nitromethane to give a red Meisenheimer-type complex which allows determination of alkylamines and quaternary ammonium compds. The mobility of the H atom (or atoms) bonded to the amino N atom of primary and secondary alkyl- and arylamines, allows derivs. which permit general or selective detns. Primary and secondary alkyl- and arylamines are estimated through the formation of N-substituted derivs. of p-nitrophenylazobenzamide or of 2,4-dinitroaniline (according to another procedure, only primary alkylamines afford the latter derivs.). Primary alkyl- and arylamines and α-amino acids react with succinic dialdehyde to give a pyrrole derivative which is then developed with p-dimethylaminobenzaldehyde. They also yield fluorescent derivs. with fluorescamine. Primary and secondary alkylamines produce fluorescent 4-amino derivs. with 7-nitrobenzofuran. Secondary alkylamines are selectively determined as N-substituted derivs. of 2-chloro-3-(2-aminoethenyl)-5,6-dicyano-1,4-benzoquinone, or of 4-amino- or 4,5-diamino-1,2-benzoquinone. Only primary arylamines condense with glutaconic dialdehyde to yield a colored Schiff's base. Diazo coupling with p-nitrophenyldiazonium ion allows the estimation of all classes of arylamines. Tertiary alkylamines and quaternary ammonium compds. develop a color with cis-aconitic anhydride in the presence of acetic anhydride, whereas only tertiary alkylamines develop a fluorescence with a mixture of aconitic acid and acetic anhydride.

ACCESSION NUMBER: 1984:465259 CAPLUS
DOCUMENT NUMBER: 101:65259
TITLE: Spectrophotometric and fluorometric determination of amines
CORPORATE SOURCE: IUPAC Analytical Chemistry Division, UK
SOURCE: Pure and Applied Chemistry (1984), 56(4), 467-77
CODEN: PACHAS; ISSN: 0033-4545
DOCUMENT TYPE: Journal
LANGUAGE: English

L6 ANSWER 14 OF 28 CAPLUS COPYRIGHT 2005 ACS on STN
AB Early stages of a photochem. reaction in a system containing a charge transfer complex were investigated. The mechanism of colored transient formation in solution and in polymeric film containing an aromatic amine-CBr2 system comprised a few steps. Time of the color stable product formation after 20 μs UV pulse depended on the nature of the aromatic amine and could reach a few seconds.

ACCESSION NUMBER: 1982:627332 CAPLUS
DOCUMENT NUMBER: 97:227332
TITLE: Early stages of the formation of colored photochemical products in polymeric and liquid media containing aromatic amines and halocarbons
AUTHOR(S): Mal'tsev, E. I.; Savel'ev, V. V.; Zolotarevskii, V. I.; Kruglov, A. B.; Vannikov, A. V.
CORPORATE SOURCE: Inst. Elektrokhim., Moscow, USSR
SOURCE: Khimiya Vysokikh Energii (1982), 16(5), 411-14
CODEN: KHVKAO; ISSN: 0023-1193
DOCUMENT TYPE: Journal
LANGUAGE: Russian

L6 ANSWER 15 OF 28 CAPLUS COPYRIGHT 2005 ACS on STN
AB Reversible thermochromic compns. contain (1) phthalein, fluorescein, or their derivative type compds. as an electron acceptor, (2) an N-containing organic compound as an electron donor, and (3) a compound which inhibits the reaction of the electron donor with the acceptor at a temperature above a certain desired temperature The thermochromic compns. are especially useful as temperature indicators. Thus, thymolphthalein 1, 1,3-diphenylguanidine 10, and stearyl alc. 100 parts were mixed to give a thermochromic composition whose color changed from blue to colorless at 50-60.

ACCESSION NUMBER: 1982:77606 CAPLUS
DOCUMENT NUMBER: 96:77606
TITLE: Reversible thermal discoloration compositions for temperature indicators
PATENT ASSIGNEE(S): Dai Nippon Printing Co., Ltd., Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 11 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 56084786	A2	19810710	JP 1979-162486	19791214
JP 61047191	B4	19861017		
PRIORITY APPLN. INFO.:			JP 1979-162486 A	19791214

L6 ANSWER 16 OF 28 CAPLUS COPYRIGHT 2005 ACS on STN
AB Acylhydrazinophenylthiourea nucleating agents having the formula RCONHNHZNHCSNR1R2 (R = H, alkyl, cycloalkyl, haloalkyl, alkoxyalkyl, phenylalkyl, or a Ph nucleus with a Hammett σ value-derived electron-withdrawing characteristic more pos. than -0.3; R1, R2 = alkyl, haloalkyl, alkoxyalkyl, phenylalkyl, cycloalkyl, a Ph nucleus with a Hammett σ value-derived electron-withdrawing characteristic less pos. than +0.50, naphthyl, or R1R2 together form a heterocyclic; Z = phenylene or alkyl-, halo-, or alkoxy-substituted phenylene). Thus, a multicolor image transfer element was prepared by coating a polyester support with a layer of gelatin and a cyan redox dye releaser; a red-sensitive internal image gelatin-AgBr emulsion layer containing Na 5-octadecylhydroquinone-2-sulfonate (I) (12 g/mol Ag) and 1-[4-(2-formylhydrazino)phenyl]-3,3-dimethylthiourea (II) (8 mg/mol Ag); an interlayer containing gelatin and didodecylhydroquinone; a layer of gelatin and a magenta redox dye releaser; a green-sensitive internal image gelatin-AgBr emulsion containing I (12 g/mol Ag) and II (10 mg/mol Ag); an interlayer of gelatin and didodecylhydroquinone; a layer containing gelatin and a yellow redox dye releaser; a blue-sensitive internal image gelatin-AgBr layer containing I (12 g/mol Ag) and II (10 mg/mol Ag); and an overcoat layer of gelatin and a latex mordant. Upon sensitometric exposure and subsequent development of this material, the blue, green, and red Dmax and corresponding Dmin values were determined to be 2.26, 2.45, and 2.40, resp., and 0.38, 0.54, and 0.35, resp., vs. 1.88, 2.15, and 0.35, resp., and 0.25, 0.34, and 0.19, resp., for a control containing 1-[4-(2-formylhydrazino)phenyl]-3-methylthiourea.

ACCESSION NUMBER: 1981:452611 CAPLUS
DOCUMENT NUMBER: 95:52611
TITLE: Acylhydrazinophenylthiourea nucleating agents and photographic emulsions and elements containing such agents
INVENTOR(S): Leone, Ronald E.
PATENT ASSIGNEE(S): USA
SOURCE: Def. Publ. U. S. Pat. Off. T, 76 pp.
CODEN: USXXBN
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 3
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 997004	H	19800805	US 1979-105317	19791219
CA 1120936	A1	19820330	CA 1979-338478	19791026
PRIORITY APPLN. INFO.:			US 1979-56588 A3	19790711

L6 ANSWER 17 OF 28 CAPLUS COPYRIGHT 2005 ACS on STN
AB MeOH stabilizes the fluorescence of carbazole (I), and the fluorescent color is affected by the addition of alkali. The fluorescent color and identification limits for compds. adsorbed on a thin-layer chromatographic (TLC) substrate are tabulated for I and its 11H-benzo(a)- (II), 5H-benzo(b)- (III), 7H-benzo(c)- (IV), 4H-benzo(def)- (V), 7H-dibenzo(cg)-(VI), 1-aza- (VII), 2-hydroxy- (VIII), and N-ethyl- (IX), derivs., iminodibenzyl (X), and 1,2-dinaphthylamine (XI). The fluorescence emission and excitation spectra and the ultraviolet absorption spectra of I-VIII in neutral and alkaline HCONMe2 are tabulated, and the fluorescent intensities in neutral and alkaline solution are compared.
The emission spectra of I and VI, the absorption spectrum of II, and the excitation spectrum of VI are reproduced. For TLC 20 + 20 cm. plates coated with Al2O3, MN-cellulose-300G, or Florisil were used. Plates were coated with Al2O3 and cellulose by the method of Brinkmann Instruments Inc. (Operating manual 103-A.), and with Florisil by mixing 35 g. with 70 ml. of H2O in a blender for 3 min. and then spreading with an applicator. Chromatographic procedures used were cellulose plates 250 μ thick developed in (A)C5H12: Et2O(19:1); (B) C5H12:CHCl3(3:2); (C) NH4OH; (D) EtOH-NH4OH; (E) cellulose plates 500 μ thick developed in 25% aqueous HCONMe2 (F) Florisil plates 500 μ thick developed in C5H12:Et2O (3:1). System A separated polynuclear hydrocarbons up to coronene; B separated carbazoles from polynuclear aromatics, aza heterocyclics, and phenols; C separated V type from other carbazoles, and by aqueous dilution of solvent from one another; D separated III from others; E separated I and V from II, III, IV, and VI; F separated as E, except that while separation of I and V from others was greater than E, separation of I from V was less. Application to the detection of III in com. pure chrysene is described. 19 references.
ACCESSION NUMBER: 1964:414819 CAPLUS
DOCUMENT NUMBER: 61:14819
ORIGINAL REFERENCE NO.: 61:2487c-f
TITLE: Fluorescent detection and spectrofluorometric characterization and estimation of carbazoles and polynuclear carbazoles separated by thin layer chromatography
AUTHOR(S): Bender, Daniel F.; Sawicki, Eugene; Wilson, Ronald M., Jr.
CORPORATE SOURCE: Robt. A. Taft Sanit. Eng. Center, Cincinnati, OH
SOURCE: Anal. Chem. (1964), 36(6), 1011-17
CODEN: ANCHAM; ISSN: 0003-2700
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L6 ANSWER 18 OF 28 CAPLUS COPYRIGHT 2005 ACS on STN
AB In the course of earlier work (CA 55, 27331g) Et 1-phenylpyrrolidine-2,5-dicarboxylate was heated with PhCH2NH2 (I) in which NaH had been dissolved. A red-purple color developed, and dibenzylamine-HCl (II) was isolated. This work was reinvestigated. NaH (52%, 2 g.) in mineral oil was added to 34 ml. I under N and the mixture warmed. The solution became pinkish at 47°, cherry red at 65°, and deep magenta at 77°; 1 ml. acid was neutralized in the receiver after 0.5 hr. at 75-7°; the temperature was kept 3.5 hrs. at 83-8°. The rate of evolution of NH3 rose to a maximum of 0.5 meq./min. after 0.5 hr. at 85°. Treatment with H2O caused loss of color. The mixture was swept 1 hr. with N, cooled, extracted with Et2O, and the extract distilled to give 11 g. II upon treatment with acid. The neutral fraction weighed 2.3 g. and had the odor of BzH. A 2nd experiment was carried out in a flask initially containing NaH suspension and evacuated to 0.04 mm.; on addition of I only a portion of the expected H was evolved, and the rest was not evolved until the temperature reached 60°. Color appeared at this point. The neutral part contained 0.7 g. BzH and PhMe Attempts to produce directed reactions using PhNH2 or PhNHMe with benzyldimethylamine were unsuccessful.
ACCESSION NUMBER: 1963:403139 CAPLUS
DOCUMENT NUMBER: 59:3139
ORIGINAL REFERENCE NO.: 59:483g-h,484a
TITLE: Displacement of ammonia from benzylamine by benzylamide anion
AUTHOR(S): Baltzly, Richard; Blackman, Samuel W.
CORPORATE SOURCE: Wellcome Res. Labs., Tuckahoe, NY
SOURCE: Journal of Organic Chemistry (1963), 28, 1158
CODEN: JOCEAH; ISSN: 0022-3263
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L6 ANSWER 19 OF 28 CAPLUS COPYRIGHT 2005 ACS on STN
AB The production of a violet color by oxidation in the presence of pyrocatechol (I) can be demonstrated with several γ of secondary amines. A pos. reaction with an optical d. of 0.3 in a 1-cm. glass cell is given by 22-60 γ Bu2NH, diethanolamine, Et2NH, piperidine, or pyrrolidine in 0.5 ml. acetone to which is added 1 ml. 0.1% I in acetone plus 2 mg. Ag2O. After 10 min. at room temperature, 2 ml. acetone is added and the color is read at 510 mμ. A similar reaction is obtained with the HCl salts of adrenalone, dibenzylamine, Bu2NH, diethanolamine, EtNH, Me2NH, ephedrine, N-methylaniline, piperidine, L(-)-proline, or pyrrolidine with 28-95 γ in 0.5 ml. H2O, to which is added 1 mol. 0.1% I in acetone, then 2 ml. acetone and approx. 2 mg. Ag2O. In this case, the reading is made at 510 mμ after 1 hr. at room temperature, except that a reaction time of 2 hrs. is required for the proline. The presence of primary amines interferes with the reaction, but tertiary amines do not react.
ACCESSION NUMBER: 1962:476365 CAPLUS
DOCUMENT NUMBER: 57:76365
ORIGINAL REFERENCE NO.: 57:15243i,15244a-b
TITLE: A color reaction of secondary amines based on formation of o-quinones
AUTHOR(S): Bartos, Jaroslav
CORPORATE SOURCE: Roussel-UCLAF, Paris
SOURCE: Ann. Pharm. Franc. (1962), 20, 478-9
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L6 ANSWER 20 OF 28 CAPLUS COPYRIGHT 2005 ACS on STN
AB Dyes of various shades, suitable for crayons, water colors, inks, pigments, and for coloring fibers such as wool, nylon, silk, are made by treating citrazinic acid (I) with amines in the presence of H2O2. Amines are RNH2 where R is a C1-18 alkyl; R'NHR'', where R' and R'' are C1-12 alkyls; XN(Y)Z, where X, Y, and Z are C1-8 alkyls. Dibenzylamine is also disclosed. The R's may contain Cl, COOH, CONH2, or up to 2 HO groups. I 15.5, and ethanolamine (II) 18.6 are heated at 50° for 12 hrs. The green color is destroyed by heating to 130°.
Stabilization is effected by neutralization with AcOH and treatment with CaCl2. The green dye is then stable to 200°. In the absence of air, no color is formed. Similarly, a blue dye was prepared from 22.5 parts 3-aminopropanol. I 15.5, dehydroabietylamine 96, iso-PrOH (91%) 250, and H2O2 (3%) 100 were heated to 90° to give a blue dye capable of forming a lacquer with Et cellulose and BuOH, giving a H2O-repellent film on fabrics. I 15.5, MeNH2 (40%) 23, H2O2 (3%) 10, and distilled H2O 10 parts are stirred at 70°. A blue dye is formed after 5 min., suitable for nylon, wool, and silk. Similarly, 59 parts Me3aN (30%) gave a blue-black dye; and 30 parts II with 40 parts concentrated HCl give a blue-green dye suitable for acetate, cotton, nylon, viscose, wool, and Orlon.
ACCESSION NUMBER: 1962:39067 CAPLUS
DOCUMENT NUMBER: 56:39067
ORIGINAL REFERENCE NO.: 56:7473e,7474a-c
TITLE: Citrazinic acid-amine-oxygen dyes
INVENTOR(S): Thomas, Frederick L.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 3000897			US	19581023

L6 ANSWER 21 OF 28 CAPLUS COPYRIGHT 2005 ACS on STN
AB cf. preceding abstract One ml. 0.1% aqueous solution of pyrazolone derivative (I) was
treated with 1 ml. 1% Mn(NO3)2, 1 drop of 1% NaOH, and, after 1-2 min. stirring, with 1-2 drops or a few crystals of organic acid; aminopyrine (II) and novalgine (III) gave intense blue colors while antipyrine gave no color. The limits (in γ) of detection for II and III with various acids were: oxalic, 50, 50; acetic, 125, 125; tartaric, 200, 200; lactic, 300, 200; citric, 300, 50. The presence of o-MeC6H4NH2 (IV) or benzidine (V) improved the sensitivity. To 5 ml. 1% Mn(NO3)2, 2 drops 20% NaOH, and a few crystals of IV or V were added, followed by 3 drops of acid after 1-2 min. stirring. Initial colors varied with the acid and I used. Colors faded or changed with time.

ACCESSION NUMBER: 1961:32150 CAPLUS
DOCUMENT NUMBER: 55:32150
ORIGINAL REFERENCE NO.: 55:6263h-i,6264a
TITLE: New color reactions of pyrazolone derivatives
AUTHOR(S): Genchev, Ml.; Pozharliev, Iv.
SOURCE: Nauch. Trudove Visshiya Med. Inst. Sofia (1959), 6(No. 1), 17-23
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L6 ANSWER 22 OF 28 CAPLUS COPYRIGHT 2005 ACS on STN
AB cf. C.A. 51, 14580g. Mg powder (2.4 g.) and 12.7 g. iodine shaken with 20 ml. anhydrous Et2O and 30 ml. anhydrous C6H6 to disappearance of the iodine color, the mixture treated with 0.1 mole Schiff base in 30 ml. anhydrous C6H6 while introducing N, the mixture shaken until all the Mg had dissolved, hydrolyzed with ice H2O, the precipitated Mg(OH)2 brought into solution
with AcOH (usually 16 g. 30% AcOH solution), the organic phase separated, the aqueous
phase extracted 2-3 times with C6H6, the combined organic phases dried, 30 ml.
Et2O added, the solution saturated with HCl, the solvents distilled, the residue
boiled a short time with Me2CO or dioxane, the extract kept overnight in the refrigerator, the precipitate filtered off, and crystallized from MeOH-Et2O gave the
ethylenediamine derivs. Products with cyclic substituents on the N atom were worked up directly by distilling the solvents and crystallizing the residue
with MeOH. The following results were obtained on reduction with Mg-MgI2 mixts. [Schiff base used, yield (g.) on working up with Me2CO, yield (g.) on working up with dioxane, product obtained, m.p., m.p. of base, nD/t given]: PhCH:NMe (I), 8.3, 6.8, (MeNHCHPh)2.2HCl, 304°, 135°, 1.5101/144-7° and 1.5203/126-8°; PhCH:NEt, 3.8, 2.1, (EtNHCHPh)2.2HCl, 261°, 86-7°, 1.5101/101-3° and 1.5203/78-9°; PhCH:NPr (II), 6.5, 3.7, (PrNHCHPh)2 (III).2HCl, 205°, 83°, 1.5000/101-2° and 1.5101/81-3°; PhCH:NCHMe2 (IV), 2.2, 2.8, (Me2CHNHCHPh)2 (V).2HCl, 250-5°, 119°, 1.4683/153-6° and 1.4840/118-20°; PhCH:NBu, -, 4.2 [direct distillation of the Et2O-C6H6 residue yielded 7.8 g.
(BuNHCHPh)2 (VI), bl 160-70°], VI.2HCl, 185-220°, oil, 1.5000/86-7° and 1.5101/76-8°; PhCH:NCH2Ph (VII), 11.9, 12.6, (PhCH2NHCHPh)2 (VIII).2HCl, 235-6°, 151°, 1.5400/168-70°, and 1.5502/145-7° (distillation of the Et2O-C6H6 residue gave VIII directly); PhCH:NCH2CH2Ph (IX), 13.5, 13.0, (PhCH2CH2NHCHPh)2 (X).2HCl, 239-40°, 123°, 1.5400/142-4° and 1.5502/115-17° (distillation of the Et2O-C6H6 residue gave 11.0 g. X directly); PhCH:NR (R = cyclohexyl) (XI), 3.6, 10.5, (RNHCHPh)2 (XII).2HCl, 261-3°, 128°, 1.5000/147-9° and 1.5101/126-7° (distillation of the Et2O-C6H6 residue gave 1.2 g. XII directly). For identification of the above compds., comparative substances were prepared by treatment of Schiff bases with activated Al according to previously described methods (loc. cit.). Analogous to previous findings, benzylalkylamines were also formed in addition to the ethylenediamines. The following results were obtained by Al reduction [Schiff base used (0.1 mole), g. substituted ethylenediamine formed, m.p., g. benzylalkylamine formed, b.p./mm., m.p. of HCl salt of benzylalkylamine given]: II, 6.2 III, 83°, 2.7 PhCH2NHPr, 102-8°/12, 184°; IV, 4.8 V, 119°, PhCH2NHCHMe2, 110-12°/12, 192°; VII, 7.7 VIII, 151°, 6.9 (PhCH2)2NH, 180°/12, 256-8°; IX, 6.1 X, 123°, PhCH2CH2NHCH2Ph, 177-9°/12, 261°; XI, 13.2 XII, 128°, PhCH2NHR, 134-7°/12, 282°. Mg powder (5.4 g.) and 50 g. iodine in 90 ml. Et2O and 90 ml. C6H6 shaken to disappearance of the iodine color, excess Mg filtered off, the filtrate treated portionwise with 48 g. I in 60 ml. C6H6, the mixture kept overnight in the refrigerator, the precipitate filtered off, washed with Et2O, and dried in a vacuum desiccator gave 84.8 g. (PhCH:NMe)2.MgI2, decomposed by H2O into I and MgI2.

L6 ANSWER 22 OF 28 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)

ACCESSION NUMBER: 1959:16976 CAPLUS
DOCUMENT NUMBER: 53:16976
ORIGINAL REFERENCE NO.: 53:3112a-g
TITLE: Reactions of Schiff bases. III. Formation of ethylenediamine derivatives from benzylidenealkylamines and magnesium-magnesium iodide mixtures
AUTHOR(S): Thies, H.; Schonenberger, H.; Bauer, K. H.
CORPORATE SOURCE: Univ. Munchen, Germany
SOURCE: Arch. Pharm. (1958), 291, 248-56
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
OTHER SOURCE(S): CASREACT 53:16976

L6 ANSWER 23 OF 28 CAPLUS COPYRIGHT 2005 ACS on STN
AB cf. C.A. 48, 2649i. The color reaction with ninhydrin and with alloxan was studied for its specificity on compds. of the ArCH(NH2)R type. The following compds. were tested: PhCH2NH2 (+, +), p- and o-CH3(C6H4)CH2NH2 (+, +), p-HOC6H4CH2NH2 (+, +), p-CH3OC6H4CH2NH2 (+, +), 3,4-(OCH2O)C6H3CH2NH2 (+, +), p- and m-HOOCC6H4CH2NH2 (+, +), p-HO3SC6H4CH2NH2 (+, +), p-NH2SO2C6H4CH2NH2 (+, +), PhCH(NH2)COOH (+, +), PhCH(NH2)CH(OH)COOH (+, +), PhCH(OH)CH(NH2)Ph (+, +), 3,4-(OCH2O)C6H3CH(OH)CH(NH2)C6H3(OCH2O)-3,4 (+, +), PhCH2NHCH3 (+, +), (PhCH2)2NH (+, +), PhCH2NHPh (-, -), p-(CH3)2NC6H4CH2NH2 (?, -), p-CH3OC6H4CH(NH2)CH(C2H5)C6H4OCH3-p (+, -), p-CH3OC6H4CH(NHCH3)CH(C2H5)C6H4OCH3-p (+, -), NH2CH2CONHCH2COOH (+, -), NH2CH2COOC2H5 (+,-), NH2CH(CH3)COOC2H5 (+, -), (CH3)2CHCH(NH2)COOC2H5(-,-), NH2C(CH3)2COOC2H5 (-, -). Pos. sign in parentheses indicates pos. reaction with ninhydrin and alloxan, resp. Moisture is necessary for the color change from yellow to purple (ninhydrin), orange to pink or purple (alloxan).

ACCESSION NUMBER: 1958:93310 CAPLUS
DOCUMENT NUMBER: 52:93310
ORIGINAL REFERENCE NO.: 52:16462h-i,16463a-b
TITLE: On the specific coloration of the benzylamine type compounds in the ninhydrin color reaction
AUTHOR(S): Takagi, I. Eiichi; Mangyo, Mitsuo; Sawai, Masanobu; Ensaka, Isao
CORPORATE SOURCE: Mitsubishi Chem. Ind. Ltd., Kanagawa
SOURCE: Bulletin of the Chemical Society of Japan (1955), 28, 213-16
CODEN: BCSJA8; ISSN: 0009-2673
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L6 ANSWER 24 OF 28 CAPLUS COPYRIGHT 2005 ACS on STN
AB cf. ibid. 943. Paper-chromatographical separation and identification were tried of 2,4'-and 4,4'-dihydroxydibenzylamine (I and II, resp.) by use of H2O and C6H6-AcOH-H2O developing agents and diazotized p-nitroaniline as color former. Neither I nor II were noticeably recognized in the paper chromatograms of resolic substances produced from HCHO and phenol in the presence of NH3 catalyst, whereas spots of I and II were clearly observed in paper chromatograms of the products by reaction between 1 mole each of phenol and HCHO in the presence of 0.05 mole (NH4)2SO4 at 50°.

ACCESSION NUMBER: 1956:72094 CAPLUS
DOCUMENT NUMBER: 50:72094
ORIGINAL REFERENCE NO.: 50:13502c-d
TITLE: 2,4'- and 4,4'-dihydroxydibenzylamine as intermediate reaction products in ammonium-catalyzed phenolic resin
AUTHOR(S): Seto, Shoji; Horiuchi, Hikaru
CORPORATE SOURCE: Osaka City Ind. Research Inst.
SOURCE: Kogyo Kagaku Zasshi (1955), 58, 987-90
CODEN: KGKZA7; ISSN: 0368-5462
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L6 ANSWER 25 OF 28 CAPLUS COPYRIGHT 2005 ACS on STN
AB Reaction between N-chloroamines and amines produces the following results: in primary and secondary aromatic amines, in which N is directly bound to the aryl group, ring chlorination takes place; in amines which have an aliphatic link to the N only exchange occurs (N-chlorination); tertiary amines (aliphatic) lose 1 alkyl group with oxidation to the aldehyde and form N-Cl derivs. N-Chloro-N-acylanilines or toluidines (unspecified) react with 2-ClOH7NH2 (equimolar amount) in C6H6 with precipitation of the base of the N-Cl derivative, while the solution gives 95-6% 1-chloro-2-naphthylamine, m. 57-8°; if an excess of chloramine is used, then in addition to precipitation of the base, there is also formed a yellow precipitate, insol. in C6H6, decompose 150°, which on warming in water or treatment with alkali turns red with loss of HCl, and becomes soluble in organic solvents; the red substance m. about 120°; their behavior suggests that the yellow solid is 1,1'-dichloro-2,7'-azonaphthalene-2HCl, while the red substance is the free azo compound; the mother liquor after removal of the ppts. yields a deep red solid, m. 108-10°, giving no m.-pt. depression with the product obtained by the above procedure. 1-ClOH7NH2 in the above reactions with an equimolar amount of N-chloroamine gave 4,1-ClClOH6NH2, m. 97° (HCl salt, m. 195°); when 2 mol of the N-chloroamine are used there is formed 2,4-dichloro-1-naphthylamine, m. 80° (HCl salt, m. 186°); 3 mol of the N-chloroamine gave a red color and HCl evolution, with separation of an amorphous dark-red solid, m. about 80°, apparently an azo derivative Equimolar amts. of N-Cl derivs. and PhZNH gave (4-ClC6H4)ZNH, m. 78°, and a crude mixture of PhZNH and Ph(4-ClC6H4) NH; 2 mol of the N-Cl derivative gave 100% of the above di-Cl derivative; 3 mol gave in addition some (2,4-Cl2C6H3)2NH, m. 135°. Addition of the N-chloramines to primary aliphatic amines gives mono-N-Cl amines in equimol. reactions and N, N-dichloroamines when 2 mol are used; the amount of active Cl in the solution does not change. Passage of dry HCl into such solns. obtained from secondary aliphatic amines results in cleavage of the R2NCl into R2NH, with formation of the original secondary amines in the form of HCl salts. Et3N with N-chloroamines gave a precipitate of the base of the chloroamine as well as an insol. precipitate, m. 235°, identified as Et3N.HCl, while the solution yields some Et2NCl, best detected by decomposition with dry HCl; in a typical experiment 10 g. Et3N gave 5.8 g. Et3N.HCl and 3.9 g. Et2NH.HCl after such treatment. PhCH2NH2 and (PhCH2)2NH react smoothly with N-chloroamines and yield N-Cl derivs. (PhCH2)3N does not appear to react on standing in C6H6 but the amount of active Cl in the solution slowly declines and a precipitate appears, identified as (PhCH2)3N.HCl, m. 227°; passage of HCl into such solution gives, among the other products, (PhCH2)2N.HCl, m. 255°; thus, 15 g. (PhCH2)3N treated as above gave 8 g. (PhCH2)3N.HCl and 5.2 g. (PhCH2)2NH.HCl, while an aqueous extract of the mixture gave 1.1 g. BzOH and some BzH. An equimol. mixture of Me2NPh and an N-chloroamine in C6H6 showed a loss of active Cl in 3-4 h. and a precipitation of the chloroamine base; the solution gave a greenish liquid, which was separated into 2 fractions, b. 206° and 232°, apparently o- and p-isomers of ClC6H4NMe2; HNO2 gave 2 NO derivs., oil and m. 55°, also characteristic of nitroso derivs. of o- and p-ClC6H4NMe2; 2 mol of N-chloroamine gave 2,4-dichlorodimethylaniline, b. 234°, while 3

L6 ANSWER 25 OF 28 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)
mol gave the 2,4,6-tri-Cl deriv., b. 247°.

ACCESSION NUMBER: 1949:36500 CAPLUS
DOCUMENT NUMBER: 43:36500
ORIGINAL REFERENCE NO.: 43:6570c-i,6571a
TITLE: Reaction of N-chloroamines with amines
AUTHOR(S): Danilov, S. N.; Koz'mina, O. P.
SOURCE: Zhurnal Obshchei Khimii (1949), 19, 309-17
CODEN: ZOKHA4; ISSN: 0044-460X
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L6 ANSWER 26 OF 28 CAPLUS COPYRIGHT 2005 ACS on STN
AB cf. C.A. 40, 7039.5. A study was made to determine whether dithiophosphinic acids are formed by a reaction analogous to that between P2S5 (I) and alcs. and phenols (cf. Cambi, C.A. 40, 3734.8), viz., by the action of P2S5 on Grignard reagents. With a suspension of P2S5 in anhydrous Et2O and MgRX (II) in the proportions represented by the ideal reaction: (1) I + 4 II → 2R2PSSMgX + MgS + MgX2, there were recovered, by decomposition of the reaction mixture by acids, RP(OH)(:S)SH (III), R2PSSH (IV), R3PS (V), and RSH (VI). VI is probably a secondary product (perhaps from II and free S), but V and III are formed by the reactions: (2) I + 6 II → 2 V + 3 MgX2 + 3MgS, and (3) I + 2 II → [RP(:S)(SMgX)]2S (VIa); VIa + 2H2O + 2HCl → 2 III + MgCl2 + H2S + MgX2. These correspond to a degree of alkylation of I greater and less, resp., than that in reaction (1), but which are completed simultaneously with the latter. Reaction (1) proceeds best at low temps. and with stoichiometric proportions, whereas reaction (3) transforms all I into phosphine sulfide only at elevated temps. and with a large excess of Grignard reagent. In no case was a quant. yield of IV obtained by reaction (1) and acidification, and under the best conditions of concentration, time, and proportions of reagents, the maximum yields were approx. 20%. Reaction (1) is recommended for the preparation of RPO(OH)2 acids, which can be obtained easily from the thio acids by oxidation with HNO3 and Br; reaction (2) is recommended for the preparation of trialkylphosphine sulfides, without passing, as do methods described in the literature, through the objectionable primary and tertiary phosphines. In brief, the reactions between I and 2, 4, and 6 mols., resp., of II lead to III, IV, and V, resp. Since in the preparation of IV, large yields of III are formed, the problem of separation is involved. This is not difficult through the Ni salts. Ni salts of IV are slightly soluble in water, and can be completely extracted by Et2O or C6H6, whereas Ni salts of III can be extracted by Et2O from aqueous solution only after acidification. Alternatively, the acid solution containing the Ni salts of III and IV can be extracted by C6H6 (which dissolves only IV salts) and then by Et2O (which dissolves III salts). I (22 g.), added slowly to 600 cc. 2 M MgEtBr (VII) in Et2O, heated 12 hrs. on a steam bath, evaporated, the residue heated 12 hrs. at 100°, 500 cc. Et2O added, excess MgEtBr decomposed by dilute H2SO4, the Et2O layer washed with dilute NaOH, evaporated at 100-10°, filtered, and the crystallized residue purified by EtOH, yields 23 g. of triethylphosphine sulfide, Et3PS (VIII), m. 94°. I (50 g.), added slowly to 600 cc. 2 M VII in Et2O, heated 12 hrs. on a steam bath, the product decomposed by water (so that acids remain as Mg salts in solution, while VIII, EtSH, and Et2S remain in the Et2O), the aqueous layer exactly neutralized, clarified by animal charcoal, acidified by dilute HCl, extracted with Et2O, the extract dried by Na2SO4, a current of dry NH3 passed through, the precipitate (the NH4 salts) dissolved in water, filtered (animal charcoal), excess NiSO4 added, extracted with C6H6, and the residue from the extract purified by EtOH and CCl4, yields 14 g. of Ni diethyldithiophosphinate, Ni(SSPEt2)2 (IX), violet, m. 110°. Treated with dilute NaOH, filtered, and extracted with Et2O, IX yields diethyldithiophosphinic acid, Et2P(:S)SH (X), an oil. By double decomposition of the NH4 salt with CdSO4, this forms the Cd salt, Cd(SSPEt2)2, m. 114°. IX and excess iodine in CCl4 or 1.5 g. X and 1.3 g.

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iodine in Et2O yield, after washing the product with dil. Na2S2O3, [Et2P(:S)S]2, a yellow oil. Comparison of IX with the Ni salt (XI) prepd. by Hofmann (Ber. 4, 430(1871)) shows the same compn. and mol. wt., but different soly., color, cryst. form, and m.p. Probably X and the acid (XII) from which H. prepd. XI represent a case of spatial isomerism with planar distribution, never observed in other compds., of the substituents around the P atom. To det. whether X can be transformed into XII, IX was kept 30 hrs. at 120°, and 6 hrs. at 150°. There was no change except incipient decompn. at 150°. Boiling 5 g. IX in C6H6 4 hrs. yielded 0.5 g. XI, but since the IX was impure, this XI may have been present originally. Furthermore, no conditions could be found in the prepn. of IX and X under which any XI or XII was formed. X (1.5 g.) in 50 cc. water, treated with 6.4 g. Br in water, filtered, evapd., taken up in 20 cc. water, excess Ag2O added, heated 6 hrs. on a steam bath, filtered, evapd., 20 cc. EtOH added, heated to the b.p., filtered, and crystd., yields Et2POOAg (cf. Ber. 25, 2439(1892)). A Ag salt with the same properties was obtained by similar oxidation of XII, but their identity could not be proved, since both decomposed before fusion. VII (200 cc. M soln. in Et2O), added dropwise to 22 g. I suspended in Et2O (heat is evolved), boiled several min., decomposed by water, the aq. layer filtered (with animal charcoal), excess aq. NiSO4 added, acidified (to Congo red), extd. with C6H6 (to remove traces of IX), the aq. soln. extd. with Et2O, the ext. evapd. on a steam bath and then in vacuo, and the residue washed with C6H6, yields Ni ethyldithiophosphonate, Ni[SSP(OH)Et]2 (XIII), violet-blue. The NH4 salt and Na salt are sol. in water (violet solns.), and, when treated with solns. of primary or secondary amines, ppt. violet cryst. Ni alkylammonium salts, e.g. the dibenzylammonium salt, (C14H16N)2Ni(S2POEt)2, the diethylammonium salt, (C4H12N)2Ni(S2POEt)2, and the diisobutylammonium salt, (C8H20N)2Ni(S2POEt)2. XIII, treated with colorless (NH4)2S, filtered, acidified, extd. with Et2O, and the ext. dried and evapd. in vacuo, yields ethyldithiophosphonic acid, EtP(OH)SSH, an oil decompg. in air (evolution of H2S). XIII, treated with Br, filtered, evapd. at 120°, NH4OH added, and evapd., yields the Ni salt, EtPO3Ni, yellow. This, treated with (NH4)2S, HNO3 added, evapd. to dryness at 250°, and the residue distd. in vacuo, yields EtPO3H2, b8 330-40°, m. 30-5° (cf. 44.5° of Hofmann, loc. cit.). iso-PrMgBr (250 cc. 2 M Et2O soln.), added slowly to 22 g. I in anhyd. Et2O, refluxed 24 hrs., decompd. by water, and the Et2O layer evapd., yields 1.5 g. of (iso-Pr)3PS. The aq. layer, neutralized (exactly to litmus), filtered with animal charcoal, acidified, extd. with Et2O, the ext. dried, dry NH32 passed through, the ppt. washed with anhyd. Et2O, dried in vacuo, dissolved in a little water, filtered with animal charcoal, concd. NiCl2 added, and the ppt. washed and dried, yields 12 g. of Ni diisopropyldithiophosphinate, [(iso-Pr)2PSS]2Ni (XIV), violet, m. 110°. This is a mixt., for fractional crystn. from EtOH yields an isomer, m. 122°, and an intense blue isomer, m. 196°. The mother liquor from the sepn. of XIV contains (iso-PrPOMgSS)2Ni, which, extd. with Et2O from the acidified soln., the ext. evapd., and the residue washed with C6H6, yields 20 g. of Ni isopropyldithiophosphonate, [iso-PrP(OH)SS]2Ni (XV), m. 167-9° (decompn.). Alc. XV (2 g.) and excess alc. HN(CH2Ph)2 (4 g.) ppt. 4.146 g. of the dibenzylammonium salt, (C14H14NH2)2(iso-PrPOSS)2Ni, of XV, violet. XV, oxidized by Br, filtered, evapd., NH4OH added, and boiled until all odor of NH3 has disappeared, yields Ni isopropylphosphonate, iso-PrPOO2Ni. Aq. XV, treated with (NH4)2S, acidified, and extd. with Et2O, yields isopropyldithiophosphonic acid, iso-PrP(OH)SSH, a yellow oil, more unstable (evolution of H2S) than X. MgPhBr (400 cc. of a 2 M soln.), added dropwise to a suspension of 22 g. I in 100 cc. anhyd. Et2O (heat is evolved), heated 12 hrs. on a steam

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bath, evapd., the residue heated several hrs. at 120°, decomposed by water, extd. with C6H6, the ext. evapd., heated to 120°, and purified by EtOH, yields 40 g. of PhPS, m. 158° (cf. 157.5° of Soden (Ann. 229, 307(1885) and 161° of Staudinger and Meyer (C.A. 14, 538)). I (22 g.) added slowly to MgPhBr (250 cc. of a 2 M soln.), heated 12 hrs. on a steam bath, evapd., heated 12 hrs. at 90-100°, 500 cc. of Et2O added to the dry residue, decomposed by water, the aq. layer neutralized (litmus), CO2 passed through to remove Et2O and H2S, filtered with animal charcoal, acidified (Congo red), extd. with Et2O, the ext. dried by Na2SO4, dry NH3 passed through, the impure NH4 salt treated with a Ni salt, and the product purified by boiling xylene, yields Ni diphenyldithiophos phinate, (Ph2PSS)2Ni (XVI), which, treated with dil. KOH, acidified, and extd. with Et2O, yields 6-8 g. of diphenyldithiophosphinic acid, Ph2PSSH, silky, m. 25-30°. The latter or XVI, oxidized by excess hot concd. HNO3, and the product purified by EtOH, yields Ph2POOH, m. 188-9° (cf. 190° of Michaelis, Ber. 12, 564(1879), and M. and Wegner, C.A. 9, 1334). I (22 g.) and MgPhBr (180 cc. of a 0.5 M soln.), agitated cold 2 hrs., heated 6-8 hrs. on a steam bath, decomposed by water, the aq. layer acidified (Congo red), extd. with Et2O, the ext. dried by Na2SO4, evapd., the residue (NH4 salt) treated with aq. NiSO4, acidified, extd. with Et2O in vacuo, and evapd. in vacuo, yield Ni phenyldithiophosphonate [PhP(OH)SS]2Ni (XVII), m. above 200° (decompn.). Phenyldithiophosphonic acid (XVIII), prepd. from XVII in the regular way, is a semisolid mass which decomposes too easily to be analyzed. Analysis of XVII showed 32% S instead of 29.35%, probably because of the presence of PhP(:S)(SH)2 (XIX), formed by hydrolysis from the presumably initial product, thus: [PhP(:S)SH]2S + H2O → XVIII + XIX. Oxidation of XIX by fuming HNO3 yields PhPO(OH)2, m. 156° (cf. 158° of Michaelis (loc. cit.) and M. and Wegner (loc. cit.)). Alc. XVII and excess alc. Et2NH give a ppt. which, filtered in vacuo, washed with EtOH, dried, yields nickel diethylammonium phenyldithiophosphinate, (C4H12N)2[PhP(:O)(:S)S]2Ni, which, by acidification and extn. with Et2O, yields XVII. Similarly XVII and HN(CH2Ph)2 form nickeldibenzylammonium phenyldithiophosphinate, (C14H9N)2[PhP(:O)(:S)S]2Ni.

ACCESSION NUMBER: 1947:9806 CAPLUS
DOCUMENT NUMBER: 41:9806
ORIGINAL REFERENCE NO.: 41:2012a-i,2013a-i,2014a-d
TITLE: The reaction between phosphorus pentasulfide and Grignard compounds
AUTHOR(S): Malatesta, Lamberto; Pizzotti, Rachele
CORPORATE SOURCE: Univ., Milano, Italy
SOURCE: Gazzetta Chimica Italiana (1946), 76, 167-81
CODEN: GCITA9; ISSN: 0016-5603
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

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AB cf. C. A. 36, 3160.6. The color test is carried out by adding 1 cc. of the organometallic solution (RLi or RMgX), without shaking, to 0.5 cc. of an approx. M solution of PhCH2NH2 or (PhCH2)2NH in unsaturate-free dry petr. ether (b. 60-8°); the appearance of a cherry-red color in a few sec. is a pos. test. If the RM solution is quite dilute, the color may fade in a few min. The shade of the red color depends to some extent on the concentration of the RM solution Amines giving a pos. test are PhCH2NH2, (PhCH2)2NH, dl-PhMeCHNH2 (pale orange in about 0.5 hr.), Ph(CH2)2NH2, Ph(CH2)3NH2 (yellow to red in 10 min.), CH2:CHCH2NH2 (orange to red in 10 min.), (CH2:CHCH2)2NH (orange to slightly red after 10 min.), PhNH2 (deep brown in 4 min.), 2-C10H7NH2, p-BrC6H4NH2 (reddish brown in 2 min.). Neg. test: (PhCH2)3N, PhCH2NMe2, MeNH2, BuNH2, Me2NH, Et2NH, HOCH2CH2NH2, PhNHMe and p-H2NC6H4NH2. Pos. tests were obtained with freshly cut Li, Na and K, RLi, RNa, EtK, Et2Sr, Et2Ba, Ph2Ba, and neg. tests with RMgX, Et2Ca, BuCaI, PhCaI and Et2Zn. Carbonation of the red solution from (PhCH2)2NH and BuLi gives 27% of α-(benzylamino)-o-toluic acid, m. 164.5-5.5°; heating at 140° gives 97.3% of the lactam, m. 89-90°.

ACCESSION NUMBER: 1943:8387 CAPLUS
DOCUMENT NUMBER: 37:8387
ORIGINAL REFERENCE NO.: 37:1397c-e
TITLE: Relative reactivities of organometallic compounds. XLV. A color test for some highly reactive organometallic compounds
AUTHOR(S): Gilman, Henry; Woods, Lauren A.
SOURCE: Journal of the American Chemical Society (1943), 65, 33-4
CODEN: JACSAT; ISSN: 0002-7863
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

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AB There is described a new reaction between alkali metals and benzylamine which is apparently given by a whole series of amines. Intensely colored compds. are formed which in certain cases can be used for the quant. detection of the presence of certain organometallic compds. The results so far obtained are reported now because of the recent appearance of a paper by Stoelzel (C. A. 35, 7381.8). It had been shown (C. A. 33, 3761.7) that Ph2C:CHNH2 (I) can be obtained from Ph2C(OH)CH2NH2 (II) with concentrated H2SO4, but the yield and purity of the product left much to be desired. In view of the extraordinary sensitivity of I to acids, it was attempted to effect the dehydration of II with a basic condensation agent. When II in toluene was refluxed with powdered NaNH2 in the absence of moisture, the individual NaNH2 particles became in a few min. an intense cornflower-blue, the solution itself remaining colorless. The color was discharged almost instantly by vigorous shaking with air, but under N it was stable. Under the same conditions Na and K instead of NaNH2 gave no color with II, but a number of amino alcs. other than II and also simple amines (none of them purely aliphatic) do form colored reaction products with NaNH2 in the absence of moisture and air. The following colors were obtained: PhCH(OH)CH(NH2)Ph, red; Ph2C(OH)CH(NH2)CH2Ph, dirty red; PhCH2CH2NH2, yellowish red; PhCH2NH2, intense red; (PhCH2)2NH, brownish red; (PhCH2)3N, red; PhNH2, dark brown; Ph2NH, dark green; Ph3N, dark green; p-toluidine, violet; p-ClC6H4NH2, brown; o-O2NC6H4NH2, red; m-O2NC6H4NH2, green; pyridine, black-brown; piperidine, red-brown. Although the color reaction is in general given by primary, secondary and tertiary aromatic and aromatic-aliphatic amines, it is possible that in individual cases the reaction of a tertiary and perhaps also of a secondary amine is due to preliminary cleavage to primary amines. The absorption spectra of the red solns. obtained from PhCH2NH2 and (PhCH2)2NH with NaNH2 were identical, but with Li instead of NaNH2 they were different. Furthermore, when (PhCH2)2NH in toluene was boiled 8 days with NaNH2 there was obtained, in addition to unchanged (PhCH2)2NH, only 0.2 g. (PhCH2)2 and no PhCH2NH2 could be detected. K, even after shaking several days, does not react with II. Later expts. showed, however, that in general all alkali metals (and also organo-alkali compds.) react but the reaction velocity depends greatly both on the concentration of the amine and on the nature of the metal. To obtain as uncomplicated a picture as possible, PhCH2NH2 was chosen for further expts. The reaction with NaNH2 is strikingly accelerated by light, the color which appears in a few min. in daylight requiring several hrs. for its development in the dark. This sensitivity to light has thus far been observed only with NaNH2 and not with Na, K or Li. The products obtained with alkali metals and with NaNH2 gave with the Zeiss step photometer curves which showed no appreciable differences. All subsequent work was done with products obtained with Li, which reacts about 10 times more rapidly than Na or K. The nature of the solvent plays but a subordinate role. A solution of PhCH2NH2 in ether with Li under N in a sealed tube attained a maximum of color in a few hrs., but after several hrs. longer the color distinctly diminished and in 24 hrs. the solution had become completely colorless and a colorless crystalline precipitate had separated In one leg of each of 4 inverted U-shaped tubes was placed a PhCH2NH2-ether-Li mixture and in the other leg ether, petr. ether, benzene and PhCH2NH2, resp., and the tubes were sealed under N. After the solns. in all 4 tubes had become colorless they were mixed with the solvents in the other leg of the tubes by tilting the tubes. In the first 3 tubes no change occurred whereas in the 4th tube the color was restored. The same effect was obtained by mere warming of the colorless solns. It has not as yet been possible to obtain the colored product in solid form

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for analysis. The colorless cryst. ppt., when removed from the N atm., immediately becomes red and in a few sec. decomps. with evolution of fumes. The fine crystals were drawn off by suction under N from the coarse particles of unchanged Li through a fine tube, then collected on an asbestos filter, washed with ether, and dried a short time in vacuo under N. The product so obtained, still moist with ether, contained N and Li in the at. ratio 1:1. Decompn. with ice water gave PhCH2NH2 and NH3 in the mol. ratio 1:1, as detd. by distn. of the volatile bases with steam, conversion into the HCl salts, evapn. and extn. with abs. alc. The Li, NH3 and PhCH2NH2 contents left 13.6% unaccounted for, in all probability due to ether still present in the original crystals. In the filtrate from the crystals, after removal of the excess of PhCH2NH2 as carbamate, were identified BzOH (with PhCH2CO2H also present), one or more amines forming no solid product with CO2, BzH and (PhCH2)2. These results indicate that the primary reaction between PhCH2NH2 and Li must be very similar (PhCH2NH2 + 2Li → PhCH2Li + LiNH2) to that between NH3 and alkali metals. To det. under what conditions the max. color intensity is obtained in the reaction, 10 and 2.5% solns. of PhCH2NH2 in ether were treated with from 1 to 1/24 equiv. of Li and the extinction coeffs. (at 458 mμ) of the mixts. were measured when the reactions had gone to completion (some days with the 10% soln., several weeks with the 2.5% soln.). The max. of extinction are obtained with a Li:PhCH2NH2 ratio of about 1:8 and are proportional to the concn. of PhCH2NH2. Although the substitution of Li for NaNH2 was already an improvement, its use still had considerable drawbacks from a preparative standpoint, and the readily available PhLi was accordingly investigated. This, too, gave a red soln. which on further addn. of PhLi was gradually decolorized and deposited a cryst. substance. This, however, was entirely different from that obtained with Li; it gave no evidence of great instability toward O and a soln. in PhCH2NH2 remained completely colorless; it dissolved easily in water without evolution of gas or any appreciable heat tone, m. 106° and had the compn. LiBr.2PhCH2NH2. On distn. in vacuo it gave pure PhCH2NH2 and left a residue of LiBr (originating from the PhLi soln., which had been prepd. from PhBr and Li in ether). Its structure was confirmed by synthesis from BuLi in benzene with PhCH2NH2.HBr and from PhCH2NH2.HBr in PhCH2NH2 with Li. Since the properties and method of prepn. of the red reaction product indicated it might be an ionized compd., cond. measurements were made under various conditions. In the mixt. of PhCH2NH2 and NaNH2 the appearance of the red color was accompanied by an appreciable cond. which disappeared with the decolorization of the soln. With Li the cond.-time curve had the same form as the curve obtained by plotting the extinction vs. the equivs. of Li (see above), showing clearly that the elec. cond. and color intensity are causally related. In measurements in which BuLi was dropped from a buret into PhCH2NH2 the cond., after reaching a max., decreased very slowly (because of the diln. by the ether of the BuLi soln.). The max. was dependent on the amt. of PhCH2NH2 and the concn. of the LiBu, lying usually in the neighborhood of 10 equivs. of LiBu; a further excess of LiBu had practically no effect. The curve obtained by adding PhCH2NH2 to BuLi was practically a straight line; a slight max. at the beginning of the curve (PhCH2NH2:LiBu = 1:1) may be due to the formation of an equimol. compd. The above reactions do not permit, as yet, any definite conclusions as to the structure of the red product or the colorless crystals. It can only be stated that the cryst. compd. corresponds in compn. to about a mol. compd. of PhCH2NHLi and NH2Li. The red color may be ascribed to soln. of PhCH2NHLi in PhCH2NH2 with formation of a complex compd. The intense colors produced by some amines with even very dil. solns. of organo-Li compds. can be utilized for the detection of organically combined Li. The amine is merely added to the

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soln. in question, which is then titrated with an approx. N soln. of EtOH in ether to disappearance of the color. Of the amines thus far studied, PhCH2NH2 and p-toluidine serve best as the indicator. The red of the PhCH2NH2 soln. changes 2 drops before the end point to a yellow color which then disappears completely. With p-toluidine, on the other hand, the soln. gradually becomes deep violet during the titration and suddenly turns at the end point to a canary-yellow which persists on further addn. of alc. Preliminary expts. indicate the method is also applicable to K and Na but not to Mg compds.

ACCESSION NUMBER: 1942:33168 CAPLUS
DOCUMENT NUMBER: 36:33168
ORIGINAL REFERENCE NO.: 36:5150h-i,5151a-i,5152a-h
TITLE: A new reaction between benzylamine and alkali metals
AUTHOR(S): Krabbe, Walter; Grunwald, Geza; Olzin, E.; Menzel, W.
SOURCE: Ber. (1941), 74B, 1343-52
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

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L8     1454481 STAB?

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SCAN -------- CC, SX, TI, ST, IT (random display, no answer numbers; SCAN must be entered on the same line as the DISPLAY, e.g., D SCAN or DISPLAY SCAN)
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FHITSTR ----- First HIT RN, its text modification, its CA index name, and its structure diagram
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              structure diagram, plus NTE and SEQ fields
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CBIB -------- AN, plus Compressed Bibliographic Data
DALL -------- ALL, delimited (end of each field identified)
DMAX -------- MAX, delimited for post-processing
FAM --------- AN, PI and PRAI in table, plus Patent Family data
FBIB -------- AN, BIB, plus Patent FAM
IND --------- Indexing data
IPC --------- International Patent Classifications
MAX --------- ALL, plus Patent FAM, RE
PATS -------- PI, SO
SAM --------- CC, SX, TI, ST, IT
SCAN -------- CC, SX, TI, ST, IT (random display, no answer numbers;
              SCAN must be entered on the same line as the DISPLAY,
              e.g., D SCAN or DISPLAY SCAN)
STD --------- BIB, IPC, and NCL

IABS -------- ABS, indented with text labels
IALL -------- ALL, indented with text labels
IBIB -------- BIB, indented with text labels
IMAX -------- MAX, indented with text labels
ISTD -------- STD, indented with text labels

OBIB -------- AN, plus Bibliographic Data (original)
OIBIB ------- OBIB, indented with text labels

SBIB -------- BIB, no citations
SIBIB ------- IBIB, no citations

HIT --------- Fields containing hit terms
HITIND ------ IC, ICA, ICI, NCL, CC and index field (ST and IT)
              containing hit terms
HITRN ------- HIT RN and its text modification
HITSTR ------ HIT RN, its text modification, its CA index name, and
              its structure diagram
HITSEQ ------ HIT RN, its text modification, its CA index name, its
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structure diagram, plus NTE and SEQ fields
FHITSTR ----- First HIT RN, its text modification, its CA index name, and its structure diagram
FHITSEQ ----- First HIT RN, its text modification, its CA index name, its structure diagram, plus NTE and SEQ fields
KWIC -------- Hit term plus 20 words on either side
OCC --------- Number of occurrence of hit term and field in which it occurs

To display a particular field or fields, enter the display field codes. For a list of the display field codes, enter HELP DFIELDS at an arrow prompt (=>). Examples of formats include: TI; TI,AU; BIB,ST; TI,IND; TI,SO. You may specify the format fields in any order and the information will be displayed in the same order as the format specification.

All of the formats (except for SAM, SCAN, HIT, HITIND, HITRN, HITSTR, FHITSTR, HITSEQ, FHITSEQ, KWIC, and OCC) may be used with DISPLAY ACC to view a specified Accession Number.
ENTER DISPLAY FORMAT (BIB):occ

L8 ANSWER 1 OF 1454481 CAPLUS COPYRIGHT 2005 ACS on STN
FIELD COUNT
AB 1

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=> d his

     (FILE 'HOME' ENTERED AT 16:14:00 ON 11 APR 2005)

     FILE 'REGISTRY' ENTERED AT 16:14:08 ON 11 APR 2005
L1             1 S DIBENZYLAMINE/CN

     FILE 'CAPLUS' ENTERED AT 16:14:43 ON 11 APR 2005
L2          1990 S 103-49-1/RN
L3        408778 S ?COLOR
L4          1791 S ?COLOUR
L5        409531 S L3 OR L4
L6            28 S L2 AND L5
L7       1658396 S PUR?
L8       1454481 S STAB?

=> s l2 and l7
L9          131 L2 AND L7

=> s l2 and l8
L10         138 L2 AND L8

=> s l9 or l10
L11         256 L9 OR L10

=> s l11 not l6
L12         243 L11 NOT L6

=> d l12 1-243 abs ibib
```

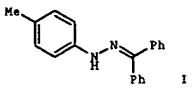
L12 ANSWER 1 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB For this study, the N'-monoamide derivs. of TTDA (3,6,10-tri(carboxymethyl)-3,6,10-triazadodecanedioic acid), N'-methylamide (TTDA-MA), N'-benzylamide (TTDA-BA), and N'-2-methoxybenzylamide (TTDA-MOBA), were synthesized. Their protonation consts. and stability consts. (log KML's) formed with Ca2+, Zn2+, Cu2+, and Gd3+ were determined by potentiometric titration in 0.10M Me4NCl at 25.0 ± 0.1°. The relaxivity values of [Gd(TTDA-MA)]-, [Gd(TTDA-BA)]-, and [Gd(TTDA-MOBA)]- remained constant with respect to pH changes over the range 4.5-12.0. The 17O NMR chemical shift of H2O induced by [Dy(TTDA-MA)(H2O)]- at pH 6.80 showed 0.9 inner-sphere H2O mols. H2O proton relaxivity values for [Gd(TTDA-MA)(H2O)]-, [Gd(TTDA-BA)(H2O)]-, and [Gd(TTDA-MOBA)(H2O)]- at 37.0 ± 0.1° and 20 MHz are 3.89, 4.21, and 4.25, resp. The H2O-exchange lifetime (τM) and rotational correlation time (τR) of [Gd(TTDA-MA)(H2O)]-, [Gd(TTDA-BA)(H2O)]-, and [Gd(TTDA-MOBA)(H2O)]- were obtained from reduced the 17O relaxation rate and chemical shifts of H217O. The 2H NMR longitudinal relaxation rates of the deuterated diamagnetic La complexes for the rotational correlation time were also thoroughly studied. The H2O-exchange rates (k298ex) for [Gd(TTDA-MA)(H2O)]-, [Gd(TTDA-BA)(H2O)]-, and [Gd(TTDA-MOBA)(H2O)]- are lower than that of [Gd(TTDA)(H2O)]2- but significantly higher than those of [Gd(DTPA)(H2O)]2- and [Gd(DTPA-BMA)(H2O)]. The rotational correlation times for [Gd(TTDA-BA)(H2O)]- and [Gd(TTDA-MOBA)(H2O)]- are significantly longer than those of [Gd(TTDA)(H2O)]2- and [Gd(DTPA)(H2O)]2- complexes. The marked increase of the relaxivity of [Gd(TTDA-BA)(H2O)]- and [Gd(TTDA-MOBA)(H2O)]- results mainly from their longer rotational correlation time. The noncovalent interaction between human serum albumin (HSA) and [Gd(TTDA-BA)(H2O)]- and [Gd(TTDA-MOBA)(H2O)]- complexes containing a hydrophobic substituent was studied by measuring the H2O proton relaxation rate of the aqueous solns. The binding association constant (KA) values are 1.0 ± 0.2 + 103 and 1.3 ± 0.2 + 103 M-1 for [Gd(TTDA-BA)(H2O)]- and [Gd(TTDA-MOBA)(H2O)]-, which indicates a stronger interaction of [Gd(TTDA-BA)(H2O)]- and [Gd(TTDA-MOBA)(H2O)]- with HSA.

ACCESSION NUMBER: 2004:1142060 CAPLUS
DOCUMENT NUMBER: 142:253131
TITLE: Synthesis and Characterization of the Novel Monoamide Derivatives of Gd-TTDA
AUTHOR(S): Wang, Yun-Ming; Li, Cha-Ru; Huang, Yu-Chin; Ou, Ming-Hung; Liu, Gin-Chung
CORPORATE SOURCE: Faculty of Medicinal and Applied Chemistry, Graduate Institute of Pharmaceutical Sciences, Kaohsiung Medical University, Kaohsiung, 807, Taiwan
SOURCE: Inorganic Chemistry (2005), 44(2), 382-392
CODEN: INOCAJ; ISSN: 0020-1669
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
REFERENCE COUNT: 47 THERE ARE 47 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 2 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

GI



I

AB The invention is directed to a process for formation of a carbon-heteroatom bond by coupling a nucleophile bearing a heteroatom susceptible of substitution with an unsatd. compound bearing a leaving group in the presence of a transition metal catalyst, a ligand (optionally), metallic hydroxides or NH4OH, and alc. as solvent. The advantages include elimination of extremely hygroscopic Na(t-OBu) and Cs2CO3 as bases, an economical and easy scale-up process. Specifically, the invention is related to arylation of nitrogen derivs., in particular hydrazones with halobenzenes in alc. solvents and phosphine ligands. For example, reacting 4-bromotoluene with benzophenone hydrazone in tert-amyl alc. in the presence of Pd(OAc)2/2-dicyclohexylphosphino-2-methylbiphenyl/NaOH at 103° for 1 h provided N-arylhydrazone I in 92% yield and 98% purity.

ACCESSION NUMBER: 2004:992725 CAPLUS
DOCUMENT NUMBER: 141:424021
TITLE: Process for formation of a carbon-heteroatom bond, in particular arylation of nitrogen-containing nucleophiles in the presence of transition metal catalysts in an alcoholic solvent
INVENTOR(S): Mauger, Christelle; Mignani, Gerard
PATENT ASSIGNEE(S): Rhodia Chimie, Fr.
SOURCE: Fr. Demande, 50 pp.
CODEN: FRXXBL
DOCUMENT TYPE: Patent
LANGUAGE: French
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
FR 2854890	A1	20041119	FR 2003-5826	20030515
WO 2004101496	A1	20041125	WO 2004-FR1159	20040512

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW
RW: BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

PRIORITY APPLN. INFO.: FR 2003-5826 A 20030515
OTHER SOURCE(S): MARPAT 141:424021
REFERENCE COUNT: 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 2 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)

L12 ANSWER 3 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB A facile preparation of a high-load, soluble oligomeric alkyl cyclohexylcarbodiimide (OACC) reagent via ROM polymerization from com. available starting materials is described. This reagent is exploited as a coupling reagent for esterification, amidation, and dehydration of carboxylic acids (aliphatic and aromatic) with an assortment of alcs. (aliphatic primary, secondary, and benzylic), thiols, phenols, and amines (aliphatic primary, secondary, benzylic, and aromatic/anilines), resp. Following the coupling event, precipitation with an appropriate solvent (Et2O, MeOH, or EtOAc), followed by filtration through a SPE provides the products in good to excellent yield and purity.

ACCESSION NUMBER: 2004:930115 CAPLUS
DOCUMENT NUMBER: 142:93482
TITLE: High-Load, Soluble Oligomeric Carbodiimide: Synthesis and Application in Coupling Reactions
AUTHOR(S): Zhang, Mianji; Vedantham, Punitha; Flynn, Daniel L.; Hanson, Paul R.
CORPORATE SOURCE: Department of Chemistry, University of Kansas, Lawrence, KS, 66045-7582, USA
SOURCE: Journal of Organic Chemistry (2004), 69(24), 8340-8344
CODEN: JOCEAH; ISSN: 0022-3263
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
REFERENCE COUNT: 43 THERE ARE 43 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 4 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB The effect of surfactants on wetting behavior of super-hydrophobic surfaces was investigated. Super-hydrophobic surfaces were prepared of alkylketene dimer (AKD) by casting the AKD melt in a specially designed mold. Time-dependent studies were carried out, using the axisym. drop shape anal. method for contact angle measurement of pure water on AKD surfaces. The results show that both advancing and receding contact angles of water on the AKD surfaces increase over time (.apprx.3 days) and reach the values of about 164 and 147°, resp. The increase of contact angles is due to the development of a prickly structure on the surface (verified by SEM), which is responsible for its super-hydrophobicity. Aqueous solns. of sodium acetate, sodium dodecyl sulfate, hexadecyltrimethylammonium bromide, and n-decanoyl-n-methylglucamine were used to investigate the wetting of AKD surfaces. Advancing and receding contact angles for various concns. of different surfactant solns. were measured. The contact angle results were compared to those of a number of pure liqs. with surface tensions similar to those of surfactant solns. It was found that although the surface tensions of pure liqs. and surfactant solns. at high concns. are similar, the contact angles are very different. Furthermore, the usual behavior of super-hydrophobic surfaces that turn super-hydrophilic when the intrinsic contact angle of liquid on a smooth surface (of identical material) is below 90° was not observed in the presence of surfactants. The difference in the results for pure liqs. and surfactant solns. is explained using an adsorption hypothesis.

ACCESSION NUMBER: 2004:804141 CAPLUS
DOCUMENT NUMBER: 142:12016
TITLE: Effect of Surfactants on Wetting of Super-Hydrophobic Surfaces
AUTHOR(S): Mohammadi, R.; Wassink, J.; Amirfazli, A.
CORPORATE SOURCE: Department of Mechanical Engineering, University of Alberta, Edmonton, AB, T6G 2G8, Can.
SOURCE: Langmuir (2004), 20(22), 9657-9662
CODEN: LANGD5; ISSN: 0743-7463
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
REFERENCE COUNT: 30 THERE ARE 30 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 5 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB Four metal complexes, [Cd(DBTC)2]2 (1), [Hg(DBTC)2] (2), [Nd(DBTC)3·2H2O] and [Nd(DBTC)3(HMPA)2] (3) (DBTC = N,N-dibenzyldithiocarbamate, HMPA = hexamethylphosphoramide), were synthesized and characterized by elemental anal. and IR spectra. The structures of complexes 1-3 were determined by X-ray crystallog. anal. Crystal data of compound 1: C30H28N2CdS4, Mr = 657.18, monoclinic, space group P21/n, a = 1.11098(4) nm, b = 1.56325(5) nm, c = 1.66695(5) nm, β = 97.9220(10)°, Z = 4, R = 0.044, wR1 = 0.091. Crystal data of compound 2: C30H28N2HgS4, Mr = 745.37, orthorhombic, space group Pbcn, a = 1.64738(1) nm, b = 1.86418(14) nm, c = 0.94000(6) nm, Z = 4, R = 0.0387, wR1 = 0.0965. Crystal data of compound 3: C57H78N9Nd02P2S6, Mr = 1319.82, monoclinic, space group P21/c, a = 1.30389(9) nm, b = 3.4708(3) nm, c = 3.1210(2) nm, β = 96.527(2)°, Z = 8, R = 0.1023, wR1 = 0.2203. Compound 1 is a dimer, and the Cd(II) ion has an approx. tetragonal pyramidal geometry. Compds. 2 and 3 are monomers and show different coordination polyhedron. The Hg(II) ion has a distorted tetrahedral coordination polyhedron, while the Nd(II) ion exhibits distorted dodecahedral geometry. Thermal gravity (TG) data indicate that compds. 1 and 2 may be sublimed, and decomposed in the course of heating and they might be expected to be useful precursors for MOCVD.

ACCESSION NUMBER: 2004:757232 CAPLUS
DOCUMENT NUMBER: 142:231750
TITLE: Synthesis, structure and thermal stability of metal complexes with N,N-dibenzyl dithiocarbamate
AUTHOR(S): Fan, Jun; Yin, Xia; Zhang, Wei-Guang; Zhang, Qi-Jiao; Lai, Chian-Sing; Tiekink, E. R. T.; Fan, Yi; Huang, Miao-You
CORPORATE SOURCE: Department of Chemistry, South China Normal University, Guangzhou, 510631, Peop. Rep. China
SOURCE: Huaxue Xuebao (2004), 62(17), 1626-1634
CODEN: HHHPA4; ISSN: 0567-7351
PUBLISHER: Kexue Chubanshe
DOCUMENT TYPE: Journal
LANGUAGE: Chinese

L12 ANSWER 6 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB The incorporation of homogeneous Ti(IV)/trialkanolamine catalyst in polymeric membranes provided new polymeric catalytic Ti(IV)-based membranes, stable and efficient as heterogeneous catalysts for chemoselective oxidns. of secondary amines to nitrones by alkyl hydroperoxides. Polyvinylidene fluoride (PVDF)-based catalytic membranes gave the best results affording products in short reaction times, high yields and selectivity using as little as 1% of catalyst, comparable with the performances of the corresponding homogeneous system. PVDF-Ti membrane could be recycled up to five runs with no loss of activity.

ACCESSION NUMBER: 2004:745016 CAPLUS
DOCUMENT NUMBER: 141:395166
TITLE: Ti(IV)-based catalytic membranes for efficient and selective oxidation of secondary amines
AUTHOR(S): Buonomenna, Maria Giovanna; Drioli, Enrico; Nugent, William A.; Prins, Leonard J.; Scrimin, Paolo; Licini, Giulia
CORPORATE SOURCE: Dip. di Ingegneria Chimica e Materiali, Universita della Calabria and ITM-CNR, Arcavacata Di Rende, I-87030, Italy
SOURCE: Tetrahedron Letters (2004), 45(40), 7515-7518
CODEN: TELEAY; ISSN: 0040-4039
PUBLISHER: Elsevier B.V.
DOCUMENT TYPE: Journal
LANGUAGE: English
REFERENCE COUNT: 21 THERE ARE 21 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 7 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB A versatile method for the synthesis of carbamates from an 'in-situ' generated polymer-supported chloroformate resin is presented. BTC (bis-trichloromethyl carbonate) is used as phosgene equivalent to afford a supported chloroformate, which, by sequential 'one-pot' reaction with a variety of alcs. and amines, furnishes the corresponding carbamates in high yields and purities.

ACCESSION NUMBER: 2004:689169 CAPLUS
DOCUMENT NUMBER: 141:349651
TITLE: A practical synthesis of carbamates using an 'in-situ' generated polymer-supported chloroformate
AUTHOR(S): Mormeneo, David; Llebaria, Amadeu; Delgado, Antonio
CORPORATE SOURCE: Facultad de Farmacia, Unidad de Quimica Farmaceutica, Universidad de Barcelona, Barcelona, 08028, Spain
SOURCE: Tetrahedron Letters (2004), 45(37), 6831-6834
CODEN: TELEAY; ISSN: 0040-4039
PUBLISHER: Elsevier B.V.
DOCUMENT TYPE: Journal
LANGUAGE: English
REFERENCE COUNT: 23 THERE ARE 23 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 8 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB α-Dibenzylamino- and α-benzyloxy- derivs. of N-acetyl-(S)-4-benzyl-5,5-dimethyloxazolidin-2-one readily undergo highly stereoselective boron mediated syn-aldol reactions with a range of aromatic and aliphatic aldehydes, generating the syn-aldol products in good to excellent yields as single diastereoisomers after purification In the α-dibenzylamino series, deprotection of the functionalized aldol fragments to the corresponding α-amino-β-hydroxy Me ester or α-amino-β-hydroxy aldehyde proved problematic, with a range of N- and O-protecting groups giving mixts. of products arising from endocyclic and exocyclic cleavage pathways. However, in the α-benzyloxy series, O-silyl protection of the aldol products, and subsequent DIBAL reduction gives stereoselectively the corresponding N-1'-hydroxyalkyloxazolidin-2-ones, which undergo base promoted fragmentation to the desired highly functionalized and differentially protected α,β-dihydroxy aldehydes in good yields and without loss of stereochem. integrity.

ACCESSION NUMBER: 2004:626631 CAPLUS
DOCUMENT NUMBER: 141:314206
TITLE: N-α-Benzyloxyacetyl derivatives of (S)-4-benzyl-5,5-dimethyloxazolidin-2-one for the asymmetric synthesis of differentially protected α,β-dihydroxy aldehydes
AUTHOR(S): Davies, Stephen G.; Hunter, Ian A.; Nicholson, Rebecca L.; Roberts, Paul. M.; Savory, Edward D.; Smith, Andrew D.
CORPORATE SOURCE: Department of Organic Chemistry, Chemistry Research Laboratory, University of Oxford, Oxford, OX1 3TA, UK
SOURCE: Tetrahedron (2004), 60(35), 7553-7577
CODEN: TETRAB; ISSN: 0040-4020
PUBLISHER: Elsevier B.V.
DOCUMENT TYPE: Journal
LANGUAGE: English
REFERENCE COUNT: 56 THERE ARE 56 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 9 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB The development of high-load, soluble oligomeric sulfonate esters, generated via ROM polymerization, and their utility in the facile benzylation of an array of amines is reported. These polymeric sulfonate esters exist as free-flowing powders, are stable at refrigerated temps., and are readily dissolved in CH2Cl2. Following the benzylation event, purification is attained via simple filtration, followed by solvent removal to deliver the desired benzylated product in good to excellent yield and high purity.

ACCESSION NUMBER: 2004:539602 CAPLUS
DOCUMENT NUMBER: 141:243951
TITLE: Development of High-Load, Soluble Oligomeric Sulfonate Esters via ROM Polymerization: Application to the Benzylation of Amines
AUTHOR(S): Zhang, Mianji; Moore, Joel D.; Flynn, Daniel L.; Hanson, Paul R.
CORPORATE SOURCE: Department of Chemistry, University of Kansas, Lawrence, KS, 66045-7582, USA
SOURCE: Organic Letters (2004), 6(16), 2657-2660
CODEN: ORLEF7; ISSN: 1523-7060
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
REFERENCE COUNT: 30 THERE ARE 30 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 10 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB Cationic complexes were designed as catalysts for imine hydrogenation processes, and it were anticipated that for this purpose naked 16e- cations or relatively labile solvent-coordinated ones possessing noncoordinating counterions would suffice. Solvento complexes [Re(CO)3(PMe3)2(S)][BArF] (4.PhCl and 4.THF) and [mer-Re(CO)2(PMe3)3(S)][BArF] (5.PhCl and 5.THF) (BArF = [B(3,5-(CF3)2C6H3)4]-; S = PhCl) were obtained from [ReH(CO)3(PMe3)2] (1) and [ReH(CO)2(PMe3)3] (2) after treatment with [Ph3C][BArF] in chlorobenzene. The five-coordinated cationic complex [Re(CO)(PMe3)4][BArF] (6) (BArF = [B(3,5-(CF3)2C6H3)4]-) was obtained by the reaction of [ReH(CO)(PMe3)4] (3) with 1 equiv of [Ph3C][BArF] in chlorobenzene. Hydride abstraction also occurred except for 1 from 2 and 3 with B(C6F5)3, producing [Re(CO)2(PMe3)3(S)][BH(C6F5)3] and [Re(CO)(PMe3)4][BH(C6F5)] (S = PhCl, THF). Treatment of ReH(CO)3(PMe3)2 (1) and ReH(CO)2(PMe3)3 (2) with 1 equiv of [isopropylisopropylideneiminium][BArF] in chlorobenzene at room temperature produced a mixture of 4.PhCl and [Re(CO)3(PMe3)2(HNiPr2)][BArF] (8) or in the case of 2 a mixture of 5.PhCl and [Re(CO)2(PMe3)3(HNiPr2)][BArF] (9) within a few minutes. After 4 h both mixts. were completely converted to 8 and 9, resp. 8 And 9 could also be obtained reacting 4.PhCl and 5.PhCl with excess diisopropylamine. Under mild conditions several imines underwent hydrogenation with H2 in the presence of 4.PhCl and 5.PhCl as catalysts. 6 Showed only poor catalysis. Further studies revealed details of the mechanism of the catalytic process. X-ray diffraction studies were carried out on the mol. structures of 4.PhCl, 5.PhCl, 6, and 5.THF.

ACCESSION NUMBER: 2004:406551 CAPLUS
DOCUMENT NUMBER: 141:150053
TITLE: Solvent Stabilization and Hydrogenation Catalysis of Trimethylphosphine-Substituted Carbonyl Rhenium Cations
AUTHOR(S): Liu, Xiang-Yang; Venkatesan, Koushik; Schmalle, Helmut W.; Berke, Heinz
CORPORATE SOURCE: Anorganisch-Chemisches Institut der Universitaet Zuerich, Zurich, CH-8057, Switz.
SOURCE: Organometallics (2004), 23(13), 3153-3163
CODEN: ORGND7; ISSN: 0276-7333
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 141:150053
REFERENCE COUNT: 68 THERE ARE 68 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 11 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB Several novel and some previously known, mostly sugar-based, surfactants have been synthesized and some of their surface properties have been characterized and compared with those of com. nonylphenol ethoxylates. The surfactant solubility in water, ethanol, and dodecane was studied. The properties of these compds. as emulsification agents in systems composed of the surfactant with water/isopropyl myristate, water/rapeseed oil, and water/dodecane are presented. The aqueous solubility of the surfactants follows the general trend expected from their hydrophilic-lipophilic balance according to Griffin (HLBG), but it is also clear that the nature of the headgroup and the structure of the nonpolar part affect the solubility in a manner not captured in the standard HLBG concept. An ester or amine group as the connecting unit between the hydrophile and the hydrophone produces a more water-soluble surfactant than the corresponding amide derivative Some effective emulsifiers were found. For instance, the surfactants with a dehydroabietic nonpolar group appear to be promising emulsifiers. Most sugar-based surfactants were able to form macro emulsions of up to around 2 wt/vol% of oil. The stability of many of these emulsions was very high, extending for months.

ACCESSION NUMBER: 2004:388282 CAPLUS
DOCUMENT NUMBER: 141:227277
TITLE: Surface properties of surfactants derived from natural products. Part 1: syntheses and structure/property relationships-solubility and emulsification
AUTHOR(S): Piispanen, Peter S.; Persson, Marcus; Claesson, Per; Norin, Torbjoern
CORPORATE SOURCE: Department of Chemistry, Organic Chemistry, Royal Institute of Technology, Stockholm, SE-100 44, Swed.
SOURCE: Journal of Surfactants and Detergents (2004), 7(2), 147-159
CODEN: JSDEFL; ISSN: 1097-3958
PUBLISHER: AOCS Press
DOCUMENT TYPE: Journal
LANGUAGE: English
REFERENCE COUNT: 44 THERE ARE 44 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 12 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB A novel safety-catch method for orthogonal synthesis of highly pure trisubstituted triazines was developed. Since the polymer-support used in this method is not acid-labile, this strategy can be uniquely applied to the synthesis of acid-sensitive triazine library compds. This method will dramatically increase the diversity of triazine and other related heterocyclic library compds.

ACCESSION NUMBER: 2004:340616 CAPLUS
DOCUMENT NUMBER: 141:38590
TITLE: Safety-Catch Approach to Orthogonal Synthesis of a Triazine Library
AUTHOR(S): Khersonsky, Sonya M.; Chang, Young-Tae
CORPORATE SOURCE: Department of Chemistry, New York University, New York, NY, 10003, USA
SOURCE: Journal of Combinatorial Chemistry (2004), 6(4), 474-477
CODEN: JCCHFF; ISSN: 1520-4766
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 141:38590
REFERENCE COUNT: 19 THERE ARE 19 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 13 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

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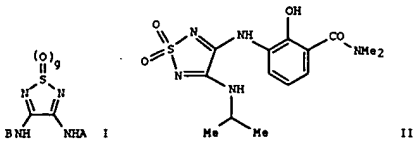

AB Disclosed are diaminothiadiazole mono- and dioxides (shown as I; e.g. II) and the pharmaceutically acceptable salts and solvates thereof. Examples of substituent A include heteroaryl, aryl, heterocycloalkyl, cycloalkyl, aryl, alkynyl, alkenyl, aminoalkyl, alkyl or amino; examples of substituent B include aryl and heteroaryl; g = 1, 2. Also disclosed is a method of treating a chemokine mediated diseases, such as, cancer, angiogenesis, angiogenic ocular diseases, pulmonary diseases, multiple sclerosis, rheumatoid arthritis, osteoarthritis, stroke and cardiac reperfusion injury, acute pain, acute and chronic inflammatory pain, and neuropathic pain using I. Although the methods of preparation are not claimed, hundreds of example prepns. and/or characterization data are included. For example, II was prepared in 31% yield from the 4-methoxy analog and isopropylamine in the presence of DIEA in MeOH; the 4-methoxy analog was prepared from the dimethoxy analog and N,N-dimethyl-3-amino-2-hydroxybenzamide in 99% crude yield. Antagonist activities of some examples of I towards CXCR1, CXCR2 and CCR7 are given.

ACCESSION NUMBER: 2004:333705 CAPLUS
DOCUMENT NUMBER: 140:357355
TITLE: Preparation of diaminothiadiazole dioxides and monoxides as CXC- and CC-chemokine receptor ligands
INVENTOR(S): Taveras, Arthur G.; Chao, Jianhua; Biju, Purakkattle J.; Yu, Younong; Fine, Jay S.; Hipkin, William; Aki, Cynthia J.; Merritt, J. Robert; Li, Ge; Baldwin, John J.; Lai, Gaifa; Wu, Minglang; Hecker, Evan A.
PATENT ASSIGNEE(S): Pharmacopeia, Inc., USA
SOURCE: PCT Int. Appl., 540 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2004033440	A1	20040422	WO 2003-US31707	20031007

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, HR, HU, ID, IL, IN, IS, JP, KG, KR, KZ, LC, LK, LR, LT, LU, LV, MA, MD, MG, MK, MN, MX, MZ, NI, NO, NZ, PG, PH, PL, PT, RO, RU, SC, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UZ, VC, VN, YU, ZA, ZM

RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY,

L12 ANSWER 13 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)

KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

US 2004186142	A1	20040923	US 2003-680393	20031007
PRIORITY APPLN. INFO.:			US 2002-417371P	P 20021009

OTHER SOURCE(S): MARPAT 140:357355
REFERENCE COUNT: 10 THERE ARE 10 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 14 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB A novel catalyst PWAA, an assembled complex of phosphotungstic acid (H3PW12O40) and a non-cross-linked copolymer of N-isopropylacrylamide with an ammonium, was developed. To this effect, N-(1-methylethyl)-2-propenamide polymer with N,N-dimethyl-N-{3-[(1-oxo-2-propenyl)amino]propyl}-1-dodecanaminium bromide was prepared and ion-exchanged with nitrate and the corresponding salt was added to phosphotungstic acid (H3PW12O40) to give the desired triphase catalyst. It is an amphiphilic, cross-linked, and supramol. insol. complex and showed catalytic activity on oxidation with aqueous hydrogen peroxide. PWAA, used in 2.7 + 10-5-2.0 + 10-3 mol equivalent, catalyzed oxidation of allylic alcs., amines, and sulfides efficiently. The turnover number (TON) of PWAA reached up to 35,000. PWAA showed a good stability in organic/aqueous media and was reused three to five times.

ACCESSION NUMBER: 2004:304411 CAPLUS
DOCUMENT NUMBER: 141:71073
TITLE: Oxidation of allylic alcohols, amines, and sulfides mediated by assembled triphase catalyst of phosphotungstate and non-cross-linked amphiphilic copolymer
AUTHOR(S): Yamada, Yoichi M. A.; Tabata, Hidetsugu; Ichinohe, Masato; Takahashi, Hideyo; Ikegami, Shiro
CORPORATE SOURCE: Faculty of Pharmaceutical Sciences, Teikyo University, Sagamiko, Kanagawa, 199-0195, Japan
SOURCE: Tetrahedron (2004), 60(18), 4087-4096
CODEN: TETRAB; ISSN: 0040-4020
PUBLISHER: Elsevier Science B.V.
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 141:71073
REFERENCE COUNT: 61 THERE ARE 61 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 15 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB A short six-step synthesis of (2S,3R,4S)-4-hydroxyisoleucine with total control of stereochem. is reported, the last step being the enzymic resolution by hydrolysis of an N-phenylacetyl lactone derivative using the com. available penicillin acylase G immobilized on Eupergit C (E-PAC).

ACCESSION NUMBER: 2004:166436 CAPLUS
DOCUMENT NUMBER: 140:357626
TITLE: Chemoenzymatic synthesis of enantiomerically pure (2S,3R,4S)-4-hydroxyisoleucine, an insulinotropic amino acid isolated from fenugreek seeds
AUTHOR(S): Rolland-Fulcrand, Valerie; Rolland, Marc; Roumestant, Marie-Louise; Martinez, Jean
CORPORATE SOURCE: Laboratoire d'Aminoacides, Peptides et Proteines, UMR - CNRS 5810 - Universite Montpellier I et II, Montpellier, 34095/5, Fr.
SOURCE: European Journal of Organic Chemistry (2004), (4), 873-877
CODEN: EJOCFK; ISSN: 1434-193X
PUBLISHER: Wiley-VCH Verlag GmbH & Co. KGaA
DOCUMENT TYPE: Journal
LANGUAGE: English
REFERENCE COUNT: 20 THERE ARE 20 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 16 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB The catalytically active orthometalated complex [Ru(phpy)(CO)2Cl]2 (phpy = phenylpyridine) was anchored to macroporous polystyrene beads through the binding of phenylpyridine moiety to the polymer backbone. The catalytic activity of the resulting species towards the reduction of organic nitro compds., alkenes, alkynes, nitriles, Schiff bases, ketones and aldehydes under high pressure, high temperature conditions in mild coordinating media was found to be comparable to that of its homogeneous analog in product selectivity but superior in stability and reusability. A tentative reduction mechanism was proposed on the basis of kinetic studies and the isolation of reactive intermediates.

ACCESSION NUMBER: 2004:138157 CAPLUS
DOCUMENT NUMBER: 141:295414
TITLE: Polystyrene anchored orthometalated ruthenium(II) complex as catalyst for the dihydrogen reduction of unsaturated organic substrates
AUTHOR(S): Islam, S. M.; Saha, C. R.
CORPORATE SOURCE: Department of Chemistry, Indian Institute of Technology, Kharagpur, 721302, India
SOURCE: Journal of Molecular Catalysis A: Chemical (2004), 212(1-2), 131-140
CODEN: JMCCF2; ISSN: 1381-1169
PUBLISHER: Elsevier Science B.V.
DOCUMENT TYPE: Journal
LANGUAGE: English
REFERENCE COUNT: 42 THERE ARE 42 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 17 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB Hydrogen-bonded phenoxyl radicals are made and the strength of the hydrogen bond between the O(phenoxyl) and the H(ammonium) atoms strongly affects their stability. The rate consts. for the intramol. proton-migration process in these systems are reported and a bifurcated hydrogen-bonded system has been characterized. Investigations show that the proton transfer from the phenoxyl-radical cation to the tertiary amine is assisted by a neighboring nitrogen atom.

ACCESSION NUMBER: 2004:132660 CAPLUS
DOCUMENT NUMBER: 140:303269
TITLE: How single and bifurcated hydrogen bonds influence proton-migration rate constants, redox, and electronic properties of phenoxyl radicals
AUTHOR(S): Thomas, Fabrice; Jarjayes, Olivier; Jamet, Helene; Hamman, Sylvain; Saint-Aman; Duboc, Carole; Pierre, Jean-Louis
CORPORATE SOURCE: Laboratoire de Chimie Biomimetique, Universite J. Fourier, Grenoble, 38041, Fr.
SOURCE: Angewandte Chemie, International Edition (2004), 43(5), 594-597
CODEN: ACIEF5; ISSN: 1433-7851
PUBLISHER: Wiley-VCH Verlag GmbH & Co. KGaA
DOCUMENT TYPE: Journal
LANGUAGE: English
REFERENCE COUNT: 26 THERE ARE 26 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 18 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB New formamidine-3TC (3TC = 2',3'-dideoxy-3'-thiacytidine) analogs have been synthesized through various methods, and their antiviral activities (HIV, HBV) have been evaluated in vitro. Anti-HIV-1 in acutely infected MT-4 cells and peripheral blood mono-cellular cells (PBMCs) showed that compds. substituted by N,N-diarylformamidine side chains at the 4-N nucleic base position (compds. 3 and 8-11) had at least equivalent anti-HIV activity as 3TC (EC50 = 0.5 and 11.6 μM, resp.). Moreover, the newly synthesized compds. demonstrated higher anti-HBV activity (EC50 ranging from 0.01 to 0.05 μM) compared to the parent nucleoside 3TC (EC50 = 0.2 μM). It should be underlined that these new promising derivs. inhibited HIV in cells of a macrophage lineage, which are known to be cellular reservoir for HIV. These results were particularly of interest, since the antiviral activities appeared not to be mediated through the formamidine bond hydrolysis and consequently the release of free 3TC. These new analog series were found to be highly stable to hydrolysis even after prolonged incubation in different biol. media (t1/2 ranged from 48 to 120 h). This enzymic stability, coupled to the fact that no delay in the antiviral response was observed compared to the free 3TC antiviral response, suggest that this new N,N-diarylformamidine nucleoside series should not be considered as classical prodrugs.

ACCESSION NUMBER: 2004:61285 CAPLUS
DOCUMENT NUMBER: 140:271129
TITLE: Potent Non-Classical Nucleoside Antiviral Drugs Based on the N,N-Diarylformamidine Concept
AUTHOR(S): Anastasi, Carole; Hantz, Olivier; De Clercq, Erik; Pannecouque, Christophe; Clayette, Pascal; Dereuddre-Bosquet, Nathalie; Dormont, Dominique; Gondois-Rey, Francoise; Hirsch, Ivan; Kraus, Jean-Louis
CORPORATE SOURCE: Laboratoire de Chimie Biomoleculaire, Developmental Biology Institute of Marseille (IBDM), Universite Mediterranee, Parc Scientifique et Technologique de Luminy, INSERM U 382, Marseille, 13288, Fr.
SOURCE: Journal of Medicinal Chemistry (2004), 47(5), 1183-1192
CODEN: JMCMAR; ISSN: 0022-2623
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
REFERENCE COUNT: 37 THERE ARE 37 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 19 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
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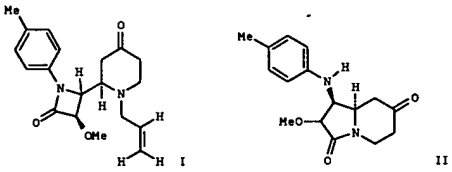

I II

AB An application of the Grubbs carbene-complex has been discovered. The catalytic deprotection of allylic amines, with reagents other than palladium catalysts, have been achieved through Grubbs carbene-mediated reaction. The catalytic system directed the reaction toward the selective deprotection of allylic amines (secondary as well as tertiary) in the presence of allylic ethers. A variety of substrates, including enantiomerically pure multifunctional piperidines, e.g., I, were also usable. This method was more convenient and chemoselective than the palladium-catalyzed method. The mechanistic hypothesis invoked a nitrogen-assisted ruthenium-catalyzed isomerization, followed by hydrolysis of the enamine intermediate. The reactive species involved in the reaction may be an Ru-H species rather than the Grubbs carbene itself. Thus, the isomerization may occur according to the hydride mechanism. The synthetic utility of this ruthenium-catalyzed allyl cleavage was illustrated by the preparation of indolizidine-type alkaloids, e.g., II.

ACCESSION NUMBER: 2004:1855 CAPLUS
DOCUMENT NUMBER: 140:181282
TITLE: Ruthenium-catalyzed chemoselective N-allyl cleavage: Novel Grubbs carbene-mediated deprotection of allylic amines
AUTHOR(S): Alcaide, Benito; Almendros, Pedro; Alonso, Jose M.
CORPORATE SOURCE: Departamento de Quimica Organica I, Facultad de Quimica, Universidad Complutense de Madrid, Madrid, 28040, Spain
SOURCE: Chemistry--A European Journal (2003), 9(23), 5793-5799
CODEN: CEUJED; ISSN: 0947-6539
PUBLISHER: Wiley-VCH Verlag GmbH & Co. KGaA
DOCUMENT TYPE: Journal
LANGUAGE: English
REFERENCE COUNT: 74 THERE ARE 74 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 20 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB Three Zn complexes with dithiocarbamate [Zn2(S2CNBu2)4](1), [Zn-(S2CNBy2)2] (2) and [Zn(S2CNBy2)2Py] (3) (By = benzyl, Py = pyridine) were synthesized. Their crystal structure, IR spectra and thermal stability were determined 1 is monoclinic, space group C2/c, with a 2.3329(3), b 1.7090(2), c 1.6115(2) nm. α 90, β 127.560(10), γ 90.°, 2 is orthorhombic, space group Pbcn, with a 1.6219(11), b 1.9001(12), c 0.9376(6) nm. α 90., β 90., γ 90.° and 3 is triclinic, space group P.hivin.1, with a 0.8642(6), b 1.3116(9), c 1.6624(11) nm. α 106.398(1), β 92.633(1), γ 107.461(1)°. 1 Is dimeric, which belongs to the typical structure of metal dithiocarbamate complexes. 2 Is monomeric which is seldom appeared in metal (except Ln, Ac series) complexes with dithiocarbamate. 2 Could coordinate with pyridine to form the five-coordinate complex 3. The center metal ion of 2 is unsatd., which is the same as some in the existed reports. The thermal stability of 1 shows that it could sublime at 251°, so 1 may be precursor for MOCVD.

ACCESSION NUMBER: 2003:984203 CAPLUS
DOCUMENT NUMBER: 140:385054
TITLE: Synthesis, structure and thermal stability of zinc complexes with dithiocarbamate
AUTHOR(S): Zhong, Yun; Zhang, Wei-Guang; Zhang, Qi-Jiao; Tan, Min-Yu; Wang, Su-Lan
CORPORATE SOURCE: Department of Chemistry, Lanzhou University, Lanzhou, 730000, Peop. Rep. China
SOURCE: Huaxue Xuebao (2003), 61(11), 1828-1833
CODEN: HHHPA4; ISSN: 0567-7351
PUBLISHER: Kexue Chubanshe
DOCUMENT TYPE: Journal
LANGUAGE: Chinese

L12 ANSWER 21 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB The systematic study of steric and electronic effects on the formation of lanthanide complexes with tridentate N,N,N',N'-tetraalkylpyridine-2,6-dicarboxamide ONO ligands (alkyl = Et: L5, isopropyl: L6 and benzyl: L7) shows a reduced affinity with increasing steric demand in the order L5 < L6 < L7. [Ln(Li)]3+ and [Ln(Li)2]3+ are formed with the three ligands, but 1:3 complexes are strictly limited to [Ln(L5)3]3+ and [Ln(L6)3]3+ because of the significant steric congestion provided by the twelve benzyl groups located along the 3-fold axis in [Ln(L7)3]3+. Comparisons between L6 and L7 in the 1:2 complexes evidence superimposable pseudo-monocapped square antiprismatic coordination spheres in the crystal structures of [Ln(Li)2(H2O)2(CF3SO3)](CF3SO3)2 (i = 6, Ln = Eu: 9; i = 7, Ln = Gd: 10). Photophys. properties of [Ln(L6)2]3+ and [Ln(L7)2]3+ (Ln = Eu, Gd, Tb, Lu) are similar except for improved quantum yields for [Ln(L7)2]3+ (Ln = Eu, Tb) which can be assigned to a slightly more efficient L7 → LnIII energy transfer process. The removal of two benzyl groups in the analogous N,N'-dibenzylpyridine-2,6-dicarboxamide ligand (L8) restores the formation of stable triple-helical complexes as demonstrated by the crystal structure of [Tb(L8)3](CF3SO3)6 (11). However, the existence of intricate mixts. of isomers in solution which are blocked on the NMR time scale limits their use as building blocks for the design of polymetallic d-f and f-f helicates.

ACCESSION NUMBER: 2003:905280 CAPLUS
DOCUMENT NUMBER: 140:103860
TITLE: Monometallic lanthanide complexes with tridentate 2,6-dicarboxamidopyridine ligands. Influence of peripheral substitutions on steric congestion and antenna effect
AUTHOR(S): Le Borgne, Thierry; Benech, Jean-Marc; Floquet, Sebastien; Bernardinelli, Gerald; Aliprandini, Christian; Bettens, Philippe; Piguet, Claude
CORPORATE SOURCE: Department of Inorganic, Analytical and Applied Chemistry, University of Geneva, Geneva, CH-1211/4, Switz.
SOURCE: Dalton Transactions (2003), (20), 3856-3868
CODEN: DTARAF; ISSN: 1477-9226
PUBLISHER: Royal Society of Chemistry
DOCUMENT TYPE: Journal
LANGUAGE: English
REFERENCE COUNT: 71 THERE ARE 71 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 22 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
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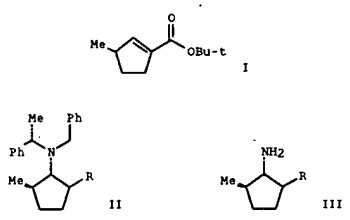

I II III

AB Conjugate addition of lithium dibenzylamide to (±)-t-butyl-3-methylcyclopentene-1-carboxylate (I) occurs with high levels of stereocontrol, with preferential addition of lithium dibenzylamide to the face of the cyclic α,β-unsatd. acceptor anti- to the 3-Me substituent. High levels of enantiorecognition are observed between I and an excess of lithium (±)-N-benzyl-N-α-methylbenzylamide (10 equivalent) (E > 140) in their mutual kinetic resolution, while the kinetic resolution of I with lithium (S)-N-benzyl-N-α-methylbenzylamide proceeds to give, at 51% conversion, (1R,2S,3R,αS)-t-butyl-3-methyl-2-N-benzyl-N-α-methylbenzylaminocyclopentane-1-carboxylate (II; R = α-CO2t-Bu) consistent with E > 130, and in 39% yield and 99 ± 0.5% de after purification Subsequent deprotection by hydrogenolysis and ester hydrolysis gives (1R,2S,3R)-3-methylcispentacin (III; R = α-CO2H) in >98% de and 98 ± 1% ee. Selective epimerization of II (R = α-CO2t-Bu) by treatment with KOtBu in tBuOH gives (1S,2S,3R,αS)-t-butyl-3-methyl-2-N-benzyl-N-α-methylbenzylaminocyclopentane-1-carboxylate (II; R = β-CO2t-Bu) in quant. yield and in >98% de, with subsequent deprotection by hydrogenolysis and ester hydrolysis giving (1S,2S,3R)-3-methyltranspentacin hydrochloride (III·HCl; R = β-CO2H)in >98% de and 97 ± 1% ee.

ACCESSION NUMBER: 2003:833184 CAPLUS
DOCUMENT NUMBER: 140:111156
TITLE: Asymmetric synthesis of (1R,2S,3R)-3-methylcispentacin and (1S,2S,3R)-3-methyltranspentacin by kinetic resolution of tert-butyl (±)-3-methylcyclopentene-1-carboxylate
AUTHOR(S): Bunnage, Mark E.; Chippindale, Ann M.; Davies, Stephen G.; Parkin, Richard M.; Smith, Andrew D.; Withey, Jonathan M.
CORPORATE SOURCE: Discovery Chemistry, IPC 675, Pfizer Global Research and Development, Kent, CT13 9NJ, UK
SOURCE: Organic & Biomolecular Chemistry (2003), 1(21), 3698-3707
CODEN: OBCRAK; ISSN: 1477-0520

L12 ANSWER 22 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)
PUBLISHER: Royal Society of Chemistry
DOCUMENT TYPE: Journal
LANGUAGE: English
REFERENCE COUNT: 59 THERE ARE 59 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 23 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB An efficient strategy for scavenging a host of nucleophiles utilizing an oligomeric bis-acid chloride (OBAC), generated from the ROM polymerization of trans-bicyclo[2.2.1]hept-5-ene-2,3-dicarbonyl dichloride, is described. The reactivity and high load of the OBAC reagent is exploited in the scavenging of amines, alcs., and thiols that are present in excess following a common benzoylation event. Following the scavenging event, these oligomers can be precipitated with EtOAc and filtered (SiO2), leaving benzoylated nucleophiles in excellent yield and purity.
ACCESSION NUMBER: 2003:829918 CAPLUS
DOCUMENT NUMBER: 140:41610
TITLE: High-Load, ROMP-Generated Oligomeric Bis-acid Chlorides: Design of Soluble and Insoluble Nucleophile Scavengers
AUTHOR(S): Moore, Joel D.; Byrne, Robert J.; Vedantham, Punitha; Flynn, Daniel L.; Hanson, Paul R.
CORPORATE SOURCE: Department of Chemistry, University of Kansas, Lawrence, KS, 66045-7582, USA
SOURCE: Organic Letters (2003), 5(23), 4241-4244
CODEN: ORLEF7; ISSN: 1523-7060
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
REFERENCE COUNT: 43 THERE ARE 43 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 24 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB The invention relates to compns. and methods for suppressing an immune response, e.g, by inhibiting class II MHC-mediated activation of T cells, to treat or prevent disorders such as rheumatoid arthritis and/or multiple sclerosis. Peptides R1-X-V-A-NRCHR2-V-NHCH[(CH2)0-1-Q-NC(:NH)NH2]-V-B-W [Q-N is pyrrolidinediyl, piperidinediyl, hexahydroazepinediyl, or octahydroazocinediyl which may be substituted by alkyl, haloalkyl, halo, OH, or amino; A is absent or is a sequence of 1-4 amino acid or amino acid analog residues; B is a sequence of 2-8 amino acid or amino acid analog residues; W is OH, alkoxy, aryloxy, or an amino group; V is CO, CS, or SO2; X is absent or is O, S, or NR; R is H or alkyl; R1, R2 are (un)substituted alkyl, heteroalkyl, alkenyl, alkynyl, aryl, aralkyl, heteroaryl, heteroaralkyl, cycloalkyl, cycloalkylalkyl, heterocyclyl, or heterocyclylalkyl; R and R2 may form a 5-7 membered ring which may be substituted or form a polycyclic structure with one or more other rings] are claimed. Thus, Ac-Cha-Gpg-Tic-Nle-βPhPro-[SΨ(oxaz)L]NMe2 [Cha = L-cyclohexylalanyl, Gpg = L-N-amidino-4-piperidinylglycyl, Tic = L-tetrahydroisoquinoline-3-carbonyl, βPhPro = 2(S),3(R)-3-phenylprolyl, [SΨ(oxaz)L] = oxazole mimetic of S-L] was prepared by the solid-phase method and its binding to MHC class II protein 0401 is shown graphically.
ACCESSION NUMBER: 2003:796420 CAPLUS
DOCUMENT NUMBER: 139:308007
TITLE: Preparation of peptides as immunosuppressants
INVENTOR(S): Nagy, Zoltan; Brandstetter, Tilmann
PATENT ASSIGNEE(S): GPC Biotech AG, Germany
SOURCE: PCT Int. Appl., 129 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2003082197	A2	20031009	WO 2003-US9219	20030324
WO 2003082197	A3	20040715		
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PH, PL, PT, RO, RU, SD, SE, SG, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW				
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				
EP 1494701	A2	20050112	EP 2003-714400	20030324
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, HU, SK				
BR 2003008654	A	20050222	BR 2003-8654	20030324

PRIORITY APPLN. INFO.: US 2002-367123P P 20020322
US 2003-367123P P 20030322
WO 2003-US9219 W 20030324

OTHER SOURCE(S): MARPAT 139:308007

L12 ANSWER 25 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
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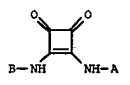

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AB Methods of treating chemokine-mediated diseases are disclosed. The methods comprise the administration of CXC-Chemokine receptor antagonists (shown as I; A = optionally substituted pyridinylalkyl, 1-oxopyridinylalkyl, thiazolylalkyl, etc.; B = optionally substituted Ph, benzotriazol-4-yl, benzimidazol-4-yl, etc.; e.g. 3-[[3-[(dimethylamino)carbonyl]-2-hydroxyphenyl]amino]-4-[[(R)-1-(5-methylfuran-2-yl)propyl]amino]cyclobutene-1,2-dione (II)), or pharmaceutically acceptable salts or solvates thereof, in combination with other classes of pharmaceutical compds. The chemokine-mediated diseases include acute and chronic inflammatory disorders, psoriasis, cystic fibrosis, asthma and cancer. Also disclosed are novel compds. I. Compds. I inhibit CXCR1 and CXCR2 chemokine receptors with IC50 <20 and <5 μM. The combination of suboptimal doses of II at 1 mg/kg (20% inhibition) and indomethacin at 0.5 mg/kg (0% inhibition) caused a significant 41% reduction of paw edema (carrageenan-induced rat paw edema model), suggesting that this combination results in greater efficacy than either agent alone. This combination did not cause a further reduction in myeloperoxidase activity in the hindpaw compared to II alone (67% inhibition for II; indomethacin = 58% inhibition; combination = 55% inhibition). The combination of suboptimal doses of II at 1 mg/kg and betamethasone at 0.05 mg/kg (32% inhibition) also demonstrated greater efficacy in inhibiting edema (61% inhibition). An additive inhibition of paw PGE2 levels was also observed (31% inhibition by either betamethasone or II alone, vs. 78% inhibition with the combination). Analogous tests were also done with the Streptococcal cell wall-induced mouse knee swelling model. Although the methods of preparation are not claimed, .apprx.50 pages of prepns. and characterization data are included.
ACCESSION NUMBER: 2003:777586 CAPLUS
DOCUMENT NUMBER: 139:291990
TITLE: Preparation of diaminocyclobutene-1,2-diones for combination treatments for chemokine-mediated diseases
INVENTOR(S): Taveras, Arthur G.; Billah, Motasim; Lundell, Daniel; Kreutner, William; Jakway, James; Fine, Jay S.; Bober, Loretta A.; Chao, Jianhua; Biju, Purakkattle; Yu, Younong
PATENT ASSIGNEE(S): Schering Corporation, USA
SOURCE: PCT Int. Appl., 214 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2003080053	A1	20031002	WO 2003-US8287	20030317
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, HR, HU,				

L12 ANSWER 25 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)

ID, IL, IN, IS, JP, KG, KR, KZ, LC, LK, LR, LT, LU, LV, MA, MD, MG, MK, MN, MX, MZ, NI, NO, NZ, PH, PL, PT, RO, RU, SC, SE, SG, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UZ, VC, VN, YU, ZA, ZM

RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

CA 2479126	AA	20031002	CA 2003-2479126	20030317
US 2004053953	A1	20040318	US 2003-390078	20030317
EP 1485089	A1	20041215	EP 2003-716685	20030317

R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, HU, SK

BR 2003008739	A	20050111	BR 2003-8739	20030317

PRIORITY APPLN. INFO.: US 2002-365314P P 20020318
WO 2003-US8287 W 20030317

OTHER SOURCE(S): MARPAT 139:291990
REFERENCE COUNT: 4 THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 26 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB Anionic polymerization initiators useful in the preparation of polymers have a protected amine functional group. The amine functionality includes a first protecting group, which can be aralkyl, Me, allyl or tertiary alkyl group. The other of the amine protecting groups can be the same as the first protecting group. Alternatively, the second protecting group can be different from the first protecting group, in which case it is selected to have differential stability to agents used to remove the aralkyl, Me, allyl or tertiary alkyl protecting group. 3-[(N-Benzyl-N-methyl)amino]-1-propyllithium was prepared and used in polymerization of isoprene.

ACCESSION NUMBER: 2003:667407 CAPLUS
DOCUMENT NUMBER: 139:197925
TITLE: Protected amino-functionalized polymerization initiators and manufacture
INVENTOR(S): Brockmann, Thorsten Werner; Hall, Randy W.
PATENT ASSIGNEE(S): FMC Corporation, USA
SOURCE: U.S., 20 pp., Cont.-in-part of U.S. 6,121,474. CODEN: USXXAM
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 3
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 6610859	B1	20030826	US 2000-665528	20000919
US 6121474	A	20000919	US 1999-256737	19990224
TW 496878	B	20020801	TW 2000-89100708	20000118
WO 2002024764	A1	20020328	WO 2001-US22911	20010719

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM

RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

AU 2001080655	A5	20020402	AU 2001-80655	20010719
GB 2382076	A1	20030521	GB 2003-3022	20010719
DE 10196639	T	20030807	DE 2001-10196639	20010719
JP 2004513087	T2	20040430	JP 2002-529172	20010719
US 2003139563	A1	20030724	US 2002-322925	20021218
US 2003162978	A1	20030828	US 2002-322926	20021218

PRIORITY APPLN. INFO.: US 1999-256737 A2 19990224
US 2000-665528 A 20000919
WO 2001-US22911 W 20010719

OTHER SOURCE(S): MARPAT 139:197925
REFERENCE COUNT: 41 THERE ARE 41 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 27 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB Magnesocene amine adducts were prepared and characterized. Addition of primary (3-amino-2,4-dimethylpentane, isopropylamine, tert-butylamine, benzylamine, cyclohexylamine) and secondary (diethylamine, dibenzylamine, dicyclohexylamine, and N-isopropylbenzylamine) amines to magnesocene at ambient temperature in toluene afforded the stable amine adducts Cp2Mg(NH2CH(CH(CH3)2)2) (91%), Cp2Mg(NH2iPr) (80%), Cp2Mg(NH2tBu) (67%), Cp2Mg(NH2CH2Ph) (80%), Cp2Mg(NH2(C6H11)) (93%), Cp2Mg(NHEt2) (84%), Cp2Mg(NH(CH2Ph)2) (86%), Cp2Mg(NH(C6H11)2) (84%), and Cp2Mg(NH(iPr)(CH2Ph)) (91%). Most adducts can be sublimed at under 100 °C/0.05 Torr in good yields (72-95%) without decomposition (<1% residue). However, Cp2Mg(NH2CH2Ph) decomps. to Cp2Mg (70% of theory) and Cp2Mg(NH2CH2Ph)2 (75% of theory) under reduced pressure, even at room temperature, and is thus unsuitable for sublimation. The solid-state structures of Cp2Mg(NH2(C6H11)), Cp2Mg(NH(iPr)(CH2Ph)), and Cp2Mg(NH2CH2Ph)2 were determined by x-ray diffraction methods. In the solid-state structures, Cp2Mg(NH2(C6H11)) and Cp2Mg(NH2CH2Ph)2 contain one η5- and one η2-coordinated cyclopentadienyl ring, while Cp2Mg(NH(iPr)(CH2Ph)) contains two η5-cyclopentadienyl rings. IR spectroscopy suggests that the adducts are stabilized by N-H···C5H-5 hydrogen bonding. MO calcns. on the model complex Cp2Mg(NH2CH3) support the idea of N-H···C5H-5 hydrogen bonding and provide insight into the energetics and exchange processes associated with the hydrogen bond. The N-H···C5H-5 hydrogen bond strength is estimated to be 4.2 ± 1.4 kcal/mol, and MO calcns. suggest that the amine hydrogen atoms undergo site exchange by a low-energy intramol. rotational process that interconverts the η2- and η5-cyclopentadienyl ligands.

ACCESSION NUMBER: 2003:664037 CAPLUS
DOCUMENT NUMBER: 139:323577
TITLE: Synthesis, Structure, and Properties of Magnesocene Amine Adducts. Structural Distortions Arising from N-H···C5H-5 Hydrogen Bonding and Molecular Orbital Calculations Thereof
AUTHOR(S): Xia, Aibing; Knox, John E.; Heeg, Mary Jane; Schlegel, H. Bernhard; Winter, Charles H.
CORPORATE SOURCE: Department of Chemistry, Wayne State University, Detroit, MI, 48202, USA
SOURCE: Organometallics (2003), 22(20), 4060-4069 CODEN: ORGND7; ISSN: 0276-7333
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 139:323577
REFERENCE COUNT: 74 THERE ARE 74 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 28 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB An effective traceless solid-phase synthesis of chlorodiaminopyrimidines via an amino-dechlorination reaction of polymer-bound 4-alkoxycarbonylamino-2,6-dichloropyrimidines has been developed. After release from the polymer the target mols. were obtained in good to excellent purity, although with modest regiocontrol. Further reaction of solid-supported N-(alkoxycarbonyl)chlorodiaminopyrimidines with secondary amines afforded triaminopyrimidines in good purity under mild conditions, whereas less nucleophilic primary amines did not perform well under the conditions explored so far.

ACCESSION NUMBER: 2003:645300 CAPLUS
DOCUMENT NUMBER: 139:292224
TITLE: Traceless solid-phase synthesis of 2,4,6-chlorodiamino- and triaminopyrimidines
AUTHOR(S): Montebugnoli, Dario; Bravo, Pierfrancesco; Brenna, Elisabetta; Mioskowski, Charles; Panzeri, Walter; Viani, Fiorenza; Volonterio, Alessandro; Wagner, Alain; Zanda, Matteo
CORPORATE SOURCE: Dipartimento di Chimica, Materiali ed Ingegneria Chimica "G. Natta", Politecnico di Milano, Milan, I-20131, Italy
SOURCE: Tetrahedron (2003), 59(36), 7147-7156 CODEN: TETRAB; ISSN: 0040-4020
PUBLISHER: Elsevier Science B.V.
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 139:292224
REFERENCE COUNT: 18 THERE ARE 18 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 29 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB Stannylalkylcarboxylate functionalized Wells-Dawson-type polyoxotungstates, α-[P2W17O61(Sn(CH2)2CO2H)]7- (I; n = 1, 2) are prepared from α-[P2W17O61]10- and ClCH2Sn(CH2)2CO2H in the presence of Bu4NBr/CH3CN. I (n = 2) reacts with primary and secondary amines, XH (e.g., XH = PhCH2NH2, (PhCH2)2NH, 1,4-NH2C6H4, NH2(CH2)5CO2H), to give α2-[P2W17O61(Sn(CH2)2CO2X)]7-.

ACCESSION NUMBER: 2003:638958 CAPLUS
DOCUMENT NUMBER: 139:307889
TITLE: Highly efficient peptide bond formation to functionalized Wells-Dawson-type polyoxotungstates
AUTHOR(S): Bareyt, Sebastian; Piligkos, Stergios; Hasenknopf, Bernold; Gouzerh, Pierre; Lacote, Emmanuel; Thorimbert, Serge; Malacria, Max
CORPORATE SOURCE: Laboratoire de Chimie Inorganique et Materiaux Moleculaires UMR 7071 CNRS, Universite Pierre et Marie Curie, Paris, 75252/05, Fr.
SOURCE: Angewandte Chemie, International Edition (2003), 42(29), 3404-3406
CODEN: ACIEF5; ISSN: 1433-7851
PUBLISHER: Wiley-VCH Verlag GmbH & Co. KGaA
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 139:307889
REFERENCE COUNT: 31 THERE ARE 31 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 30 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

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* STRUCTURE DIAGRAM TOO LARGE FOR DISPLAY - AVAILABLE VIA OFFLINE PRINT *

AB Ansamycins of formula I [R1R2 = H2, bond; R3 = H, alkyl; R4, R5 = H, OH, alkoxy, acetoxy, aryloxy, acyloxy, etc.; R4R5 = O, NOH, alkoxyimine, etc.; R6 = H, alkyl, aryl, acyl; Y1, Y2 = H, OH, alkoxy, acetoxy, acyloxy, alkylsulfonyl, alkylamino, etc.; Y1R4 = heterocyclic or carbocyclic ring] and methods of preparing and using the same are described. At least some of these ansamycins exhibit one or more of improved aqueous formulation ability, chemical stability, and bioavailability. Some of the derivs. described are dimers. These and others described can include one or more solubilizing groups that have expected merit in rendering the overall compds. useful as drugs and prodrugs. Thus, II was prepared from geldanamycin and 3,3'-diaminodipropylamine in 93% yield. II suppressed tumor growth of BT474 and SKOV-3 tumor models.

ACCESSION NUMBER: 2003:633428 CAPLUS
DOCUMENT NUMBER: 139:164658
TITLE: Preparation of ansamycins having improved pharmacological and biological properties
INVENTOR(S): Zhang, Lin; Le Brazidec, Jean-Yves; Boehm, Marcus F.; McHugh, Sean Konrad; Fan, Junhua; Fritz, Lawrence C.; Burrows, Francis J.
PATENT ASSIGNEE(S): Conforma Therapeutics Corporation, USA
SOURCE: PCT Int. Appl., 207 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 2
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2003066005	A2	20030814	WO 2003-US4283	20030210
WO 2003066005	A3	20040610		
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW				
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				
WO 2003050295	A2	20030619	WO 2002-US39993	20021212
WO 2003050295	A3	20050210		
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL, PT, RO, RU, SD, SE, SG, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW				
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES,				

L12 ANSWER 30 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)

FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

EP 1472230 A2 20041103 EP 2003-713437 20030210

R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, HU, SK

PRIORITY APPLN. INFO.:

US 2002-355275P	P	20020208
US 2002-367055P	P	20020322
WO 2002-US39993	A	20021212
US 2001-340762P	P	20011212
WO 2003-US4283	W	20030210

OTHER SOURCE(S): MARPAT 139:164658

L12 ANSWER 31 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB A novel, mild method for the synthesis of disubstituted and trisubstituted N-acyl ureas on solid support is described. Addition of carboxylic acids to a resin-bound carbimidoyl chloride gave, initially, an O-acyl isourea which subsequently rearranged to the corresponding N-acyl urea. Trisubstituted N-acyl ureas were assembled on a Wang resin from a wide range of Fmoc amino acids, secondary amines and carboxylic acids. Acid mediated cleavage yielded the products in good yields and excellent purities. In addition, the regioselective synthesis of disubstituted N-acyl ureas is demonstrated with four examples. Compds. thus prepared included 4-[[[benzoyl(1-piperidinylcarbonyl)]amino]methyl]benzeneacetic acid, 3-(benzoyl(1-piperidinylcarbonyl)amino]benzoic acid, 4-[benzoyl(1-piperidinylcarbonyl)amino]butanoic acid, 4-[[[(cyclohexylcarbonyl)(1-piperidinylcarbonyl]amino]methyl]benzeneacetic acid, 4-[[[benzoyl[(phenylamino)carbonyl]amino]methyl]benzeneacetic acid.

ACCESSION NUMBER: 2003:627048 CAPLUS
DOCUMENT NUMBER: 139:337930
TITLE: A novel solid-phase synthesis of di- and trisubstituted N-acyl ureas
AUTHOR(S): Ravn, Jacob; Ankersen, Michael; Begtrup, Mikael; Lau, Jesper F.
CORPORATE SOURCE: Medicinal Chemistry, Novo Nordisk A/S, Maaloev, DK-2760, Den.
SOURCE: Tetrahedron Letters (2003), 44(36), 6931-6935
CODEN: TELEAY; ISSN: 0040-4039
PUBLISHER: Elsevier Science B.V.
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 139:337930
REFERENCE COUNT: 12 THERE ARE 12 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 32 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB N2O4 was supported on cross-linked polyvinylpyrrolidone to afford a solid, stable and recyclable nitrosation agent. This reagent showed excellent selectivity for N-nitrosation of dialkyl amines in the presence of diaryl-, aralkyl-, trialkylamines, and also for secondary amides under mild and heterogeneous conditions. Also N-nitroso-N-alkylamides were selectively prepared in the presence of primary amides and N-phenylamides under similar reaction conditions. Selective N-nitrosation or dealkylation and N-nitrosation of tertiary amines was also performed by this reagent.

ACCESSION NUMBER: 2003:608957 CAPLUS
DOCUMENT NUMBER: 140:59602
TITLE: Selective N-nitrosation of amines, N-alkylamides, and N-alkylureas by N2O4 supported on cross-linked polyvinylpyrrolidone (PVP-N2O4)
AUTHOR(S): Iranpoor, Nasser; Firouzabadi, Habib; Pourali, Ali-Reza
CORPORATE SOURCE: Department of Chemistry, Shiraz University, Shiraz, 71454, Iran
SOURCE: Synthesis (2003), (10), 1591-1597
CODEN: SYNTBF; ISSN: 0039-7881
PUBLISHER: Georg Thieme Verlag
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 140:59602
REFERENCE COUNT: 74 THERE ARE 74 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 33 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB Disclosed is a method for producing a nitrone compound or an N-oxyl compound, characterized in that it comprises reacting a secondary amine and hydrogen peroxide in the presence of a metal oxide catalyst formed by reacting hydrogen peroxide with at least one selected from the group consisting of metallic tungsten, metallic molybdenum, a tungsten compound comprising tungsten and an element belonging to Group IIIb, Group IVb, Group Vb, or Group VIb except oxygen, and a molybdenum compound comprising molybdenum and an element belonging to Group IIIb, Group IVb, Group Vb, or Group VIb except oxygen. Thus, 160 mg tungsten metal and 2.5 g aqueous 30 weight% H2O2 were added to a 50 mL flask, heated to 40°, stirred at the same temperature for 0.5 h to prepare an. aqueous solution of tungsten oxide which was cooled to 20°, treated with 30 g H2O and 1.7 g 1,2,3,4-tetrahydroisoquinoline, and then dropwise with 6.9 g aqueous 30 weight% H2O2 over 30 min, stirred at the same temperature for 3 h, treated with 50 g Me tert-Bu ether and 10 g H2O, stirred at room temperature, and left to stand for phase separation, followed by concentration of the organic layer to give 2.1 g 3,4-dihydroisoquinoline N-oxide as a light yellow oil (80% purity based on GC anal., 90% yield).

ACCESSION NUMBER: 2003:591141 CAPLUS
DOCUMENT NUMBER: 139:149534
TITLE: Method for producing nitrone compound and N-oxyl compound
INVENTOR(S): Hagiya, Koji
PATENT ASSIGNEE(S): Sumitomo Chemical Company, Limited, Japan
SOURCE: PCT Int. Appl., 23 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2003062193	A1	20030731	WO 2003-JP243	20030115
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, KE, KG, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW				
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				
JP 2003286242	A2	20031010	JP 2002-354780	20021206
JP 2004149513	A2	20040527	JP 2003-207088	20030811
PRIORITY APPLN. INFO.:			JP 2002-15300 A	20020124
			JP 2002-256424 A	20020902

OTHER SOURCE(S): CASREACT 139:149534; MARPAT 139:149534
REFERENCE COUNT: 22 THERE ARE 22 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 34 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB Solid-supported barbituric acid can be used for the palladium(0)-catalyzed deprotection of allyl amines, carbamates, carbonates, esters and ethers. This solid-supported reagent facilitates isolation and purification of the deprotected compds., especially acids and amines.

ACCESSION NUMBER: 2003:513197 CAPLUS
DOCUMENT NUMBER: 139:307359
TITLE: Facile removal strategy for allyl and allyloxycarbonyl protecting groups using solid-supported barbituric acid under palladium catalysis
AUTHOR(S): Tsukamoto, Hirokazu; Suzuki, Takamichi; Kondo, Yoshinori
CORPORATE SOURCE: Graduate School of Pharmaceutical Sciences, Tohoku University, Sendai, 980-8578, Japan
SOURCE: Synlett (2003), (8), 1105-1108
CODEN: SYNLES; ISSN: 0936-5214
PUBLISHER: Georg Thieme Verlag
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 139:307359
REFERENCE COUNT: 16 THERE ARE 16 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 35 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB A symposium report. Amino acids and peptides (S)-R1NHCHR2CO2H [R1 = Boc, Z, Boc-Ile, Bos-Lys(2-ClZ), Boc-Pro, Fmoc-Ile; R2 = CH2OCH2Ph, CH2Ph, (S)-CHMe2, (R)-CHMe2, CHMe2, CH2CHMe2] were converted to the O-succinimidyl carbamates R1NHCHR2NHCO2Su (I). I are stable and can be stored without any degradation I are novel building blocks for the efficient solution synthesis of ureidopeptides and peptidyl hydantoins and for the solid-phase synthesis of oligourea/peptide hybrids.

ACCESSION NUMBER: 2003:509493 CAPLUS
DOCUMENT NUMBER: 140:199685
TITLE: Solution and solid-phase synthesis of ureidopeptides and oligourea/peptide hybrids
AUTHOR(S): Semetey, Vincent; Schaffner, Arnaud-Pierre; Briand, Jean-Paul; Guichard, Gilles
CORPORATE SOURCE: Laboratoire de Chimie Immunologique, CNRS UPR 9021, IBMC, Strasbourg, 67084, Fr.
SOURCE: Peptides 2000, Proceedings of the European Peptide Symposium, 26th, Montpellier, France, Sept. 10-15, 2000 (2001), Meeting Date 2000, 273-274. Editor(s): Martinez, Jean; Fehrentz, Jean-Alain. Editions EDK: Paris, Fr.
CODEN: 69EDWK; ISBN: 2-84254-048-4
DOCUMENT TYPE: Conference
LANGUAGE: English
REFERENCE COUNT: 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 36 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB A 1,3-diketone resin was developed as the basis for a selective scavenger for hydrazines. In addition, it can be employed for the selective removal of primary amines in the presence of secondary amines which is of fundamental importance in the purification of reductive alkylations. The resin's specificity is based on the sequestration of the hydrazine via their polymer-attached pyrazoles and of the primary amines via their enamines.

ACCESSION NUMBER: 2003:468746 CAPLUS
DOCUMENT NUMBER: 139:337915
TITLE: A polymer-bound 1,3-diketone: A highly efficient scavenger for hydrazines, and primary amines
AUTHOR(S): Schoen, Uwe; Messinger, Josef; Mersyo, Nuria; Juszkiewicz, Grzegorz; Kirschning, Andreas
CORPORATE SOURCE: Solvay Pharmaceuticals GmbH, Hannover, 30173, Germany
SOURCE: Synlett (2003), (7), 983-986
CODEN: SYNLES; ISSN: 0936-5214
PUBLISHER: Georg Thieme Verlag
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 139:337915
REFERENCE COUNT: 20 THERE ARE 20 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 37 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB Benzotriazole-1-carboxamide is a new efficient reagent for the preparation of mono- and N,N-disubstituted ureas. The title ureas R1NR2CONH2 (R1 = p-MeOC6H4, PhCH2, pentyl, etc.; R2 = H, Bu, PhCH2, Me2CH) were obtained from benzotriazole-1-carboxamide with primary and secondary aliphatic amines R1R2NH and p-anisidine under mild conditions with simple purification in isolated yields of 61-96%. The procedure developed is suitable for solid-phase work.

ACCESSION NUMBER: 2003:459554 CAPLUS
DOCUMENT NUMBER: 140:128130
TITLE: Synthesis of mono- and N,N-disubstituted ureas
AUTHOR(S): Katritzky, Alan R.; Kirichenko, Nataliya; Rogovoy, Boris V.
CORPORATE SOURCE: Center for Heterocyclic Compounds, Department of Chemistry, University of Florida, Gainesville, FL, 32611-7200, USA
SOURCE: ARKIVOC (Gainesville, FL, United States) (2003), (8), 8-14
CODEN: AGFUAR
URL: http://www.arkat-usa/org/ark/journal/2003/Fukumoto/KF-627H/627H.pdf
PUBLISHER: Arkat USA Inc.
DOCUMENT TYPE: Journal; (online computer file)
LANGUAGE: English
REFERENCE COUNT: 34 THERE ARE 34 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 38 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB Cu(II) dialkyldithiocarbamate complexes, Cu(S2CNRR')2, with R = R' = Bu (1); i-Bu (2); c-Hex (3); CH2Ph (4); R = Bu, R' = Et (5); R = Pr, R' = c-PrCH2 (6); R = R' = Pr (7); i-Pr (8); allyl (9), were prepared The thermal properties of the complexes were studied to determine if their potential performance in CVD processes was affected by the nature of the peripheral substituents of the ancillary ligands. Modest gains in volatility were noted for 2 and 7 over the most often used complex with R = R' = Et, while 1 and 8 had thermal parameters and stability comparable to this standard Unsym. substitution, such as in 5, also improved volatility, with some loss of stability for this particular compound X-ray diffraction studies of complexes 1-6 suggested that long range Cu···S interactions in the solid-state have little bearing on the thermal properties of this class of Cu(II) complexes.

ACCESSION NUMBER: 2003:445282 CAPLUS
DOCUMENT NUMBER: 139:344750
TITLE: Thermal and structural characterization of a series of homoleptic Cu(II) dialkyldithiocarbamate complexes: bigger is only marginally better for potential MOCVD performance
AUTHOR(S): Ngo, Silvana C.; Banger, Kulbinder K.; DelaRosa, Mark J.; Toscano, Paul J.; Welch, John T.
CORPORATE SOURCE: Department of Chemistry, The University at Alban State University of New York, Albany, NY, 12222, USA
SOURCE: Polyhedron (2003), 22(12), 1575-1583
CODEN: PLYHDE; ISSN: 0277-5387
PUBLISHER: Elsevier Science Ltd.
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 139:344750
REFERENCE COUNT: 46 THERE ARE 46 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 39 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB The present invention discloses an improved method for the manufacture of Pravastatin sodium salt by fermentation under optimal fermentation parameters using a new strain of Streptomyces flavidovirens. Specifically, Streptomyces flavidovirens BICC 6826 (DSM 14455) can regioselectively hydroxylate the pravastatin precursor compactin at the 6β position. Thus, Streptomyces flavidovirens BICC 6826 was grown in fed-batch fermentation mode where the feed consisted of compactin or a compactin salt and/or dextrose. The fermentation was conducted at pH 7.6-8.0 and 28 °C. The resulting sodium pravastatin salt was then harvested and purified with a variety of techniques.

ACCESSION NUMBER: 2003:261993 CAPLUS
DOCUMENT NUMBER: 138:270408
TITLE: Process for producing pravastatin sodium salt using Streptomyces flavidovirens DSM 14455
INVENTOR(S): Gururaja, Ramavana; Goel, Anuj; Sridharan, Madhavan; Melarkode, Ramakrishnan Sadhana; Kulkarni, Madhav; Poornaprajna, Acharya; Sathyanathan, Deepthy; Ganesh, Sambasivam; Suryanarayan, Shrikumar
PATENT ASSIGNEE(S): Biocon India Limited, India
SOURCE: PCT Int. Appl., 18 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2003027302	A1	20030403	WO 2001-IN161	20010927
WO 2003027302	C2	20030515		
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW				
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				
EP 1430138	A1	20040623	EP 2001-976603	20010927
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR				
BR 2001017138	A	20041013	BR 2001-17138	20010927
JP 2005503174	T2	20050203	JP 2003-530867	20010927
US 2004209335	A1	20041021	US 2004-485782	20040204
PRIORITY APPLN. INFO.:			WO 2001-IN161 W	20010927

REFERENCE COUNT: 6 THERE ARE 6 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 40 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB Dihydrogen reduction of aliphatic and aromatic nitrocompounds, alkenes, alkynes, nitriles and Schiff bases to their corresponding saturated products is efficiently carried out using the soluble and polymer anchored palladium (II) complexes. The immobilization of the palladium (II) complexes in the polymer matrix slightly decreased the catalytic activities on the basis of metal content but improved the thermal and chemical stabilities and product selectivities relative to those of the corresponding homogeneous ones. The soluble catalyst has the propensity to decompose under high pressure, high temperature conditions but the immobilized ones can be used repeatedly and can be stored for long periods without any appreciable loss of catalytic activity. XPS study indicates the presence of palladium (II) in the fresh and used catalyst and a plausible reaction mechanism has been suggested on the basis of exptl. findings.

ACCESSION NUMBER: 2003:155486 CAPLUS
DOCUMENT NUMBER: 138:387114
TITLE: Polymer supported palladium (II) complexes as hydrogenation catalysts
AUTHOR(S): Mukherjee, Debkumar
CORPORATE SOURCE: Department of Chemistry, Ramsaday College, Howrah, 711 401, India
SOURCE: Indian Journal of Chemistry, Section B: Organic Chemistry Including Medicinal Chemistry (2003), 42B(2), 346-352
CODEN: IJSBDB; ISSN: 0376-4699
PUBLISHER: National Institute of Science Communication
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 138:387114
REFERENCE COUNT: 30 THERE ARE 30 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 41 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB Amines are manufactured by the reaction of aldehydes or ketones with NH3 or primary or secondary amines in the presence of a H-donor and of homogeneous metal catalysts of the VIII-subgroup, under mild conditions. For example, stirring a mixture of 240 mg PhCOMe, 0.63 g HCO2NH4, 40 mg [Ru[(R)-TolBINAP](DMF)xCl2] complex catalyst [(R)-TolBINAP = (R)-2,2'-bis(di-p-tolylphosphino)-1,1'-binaphthyl] and 4 mL of 20% ammonia solution for 16 h at 100° gave a mixture of 96% (R)-1-phenylethylamine (optical purity 93%) and 4% PhCHMeOH.

ACCESSION NUMBER: 2003:133220 CAPLUS
DOCUMENT NUMBER: 138:189782
TITLE: Manufacture of amines by reductive amination of carbonyl compounds under transfer-hydrogenation conditions
INVENTOR(S): Boerner, Armin; Dingerdissen, Uwe; Kadyrov, Renat; Riermeier, Thomas; Tararov, Vitali
PATENT ASSIGNEE(S): Degussa AG, Germany
SOURCE: PCT Int. Appl., 47 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2003014061	A1	20030220	WO 2002-EP8748	20020806
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZM, ZW				
RW: AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, SK, TR				
DE 10138140	A1	20030220	DE 2001-10138140	20010809
EP 1414783	A1	20040506	EP 2002-767327	20020806
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, SK				
JP 2004537588	T2	20041216	JP 2003-519013	20020806
US 2004267051	A1	20041230	US 2004-484900	20040818
PRIORITY APPLN. INFO.:			DE 2001-10138140 A	20010809
			WO 2002-EP8748 W	20020806

OTHER SOURCE(S): MARPAT 138:189782
REFERENCE COUNT: 3 THERE ARE 3 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 42 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB The regeneration and reuse of a supported 4-hydroxybenzaldehyde scavenger (I) for amine sequestration has been achieved up to three times without significant loss of activity. The scavenging process between the aldehyde resin I and a range of amines has been investigated in detail to determine the scope of this scavenger. Its application for the rapid purification of a small library of secondary amines has also been demonstrated, and it has been shown that the large excess of scavenger resin used can be recovered and recycled, making this a more cost-effective process.

ACCESSION NUMBER: 2003:45383 CAPLUS
DOCUMENT NUMBER: 138:221043
TITLE: Recycling and Reuse of a Polymer-Supported Scavenger for Amine Sequestration
AUTHOR(S): Guino, Meritxell; Brule, Emilie; de Miguel, Yolanda R.
CORPORATE SOURCE: Department of Chemistry, King's College London, London, WC2R 2LS, UK
SOURCE: Journal of Combinatorial Chemistry (2003), 5(2), 161-165
CODEN: JCCHFF; ISSN: 1520-4766
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 138:221043
REFERENCE COUNT: 31 THERE ARE 31 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 43 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB This paper reports the formation of novel hydrogen-bonded assemblies 13·CA obtained upon mixing cyanuric acid (CA) with melamine derivs. 1, in which two of the three possible H-bonding arrays have been blocked. The four components are held together by 9 hydrogen bonds and form a rigid planar structure in which a central CA (three ADA motifs: A = acceptor, D = donor) is hydrogen bonded to three peripheral melamine derivs. (DAD motif). Furthermore, the synthesis and assembly studies are described of hydrogen-bonded assemblies 2·4·CA, comprised of three melamine derivs. that are covalently connected, and CA. The overall thermodn. stability of assemblies 2·4·CA is superior to 13·CA (ITm = 9 vs 3.6). The presence of the 2·CA complex in chloroform was confirmed by 1H NMR spectroscopy and MALDI-TOF mass spectromstry. Substitution of the trimelamines with chiral or fluorescent groups (R3) enabled the study of the assemblies by CD and fluorescence spectroscopy. Titration expts. revealed strongly enhanced stabilities even in the presence of polar solvents, such as THF and CH3OH. Depending on the polarity of the solvent, stacking between the planar assembly units was observed

ACCESSION NUMBER: 2003:20468 CAPLUS
DOCUMENT NUMBER: 138:187358
TITLE: A Novel Type of Hydrogen-Bonded Assemblies Based on the Melamine·Cyanuric Acid Motif
AUTHOR(S): Arduini, Maria; Crego-Calama, Mercedes; Timmerman, Peter; Reinhoudt, David N.
CORPORATE SOURCE: Laboratory of Supramolecular Chemistry and Technology, MESA+ Research Institute, University of Twente, Enschede, 7500 AE, Neth.
SOURCE: Journal of Organic Chemistry (2003), 68(3), 1097-1106
CODEN: JOCEAH; ISSN: 0022-3263
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 138:187358
REFERENCE COUNT: 40 THERE ARE 40 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 44 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB A library of triamino-1,3,5-triazines are prepared on solid-phase using the oxidation of benzylthiotriazines to benzylsulfonyltriazines followed by nucleophilic substitution of the benzylsulfonyltriazines with amines as the key steps. Attachment of a primary amine to a formyl-substituted polystyrene (PAL) resin, addition of a dichloro(benzylthio)-1,3,5-triazine to the resin-bound primary amine, substitution of the chlorine atom with an amine, oxidation of the benzylthio moiety, substitution of the newly generated benzylsulfonyl moiety with a second amine, and resin cleavage with trifluoroacetic acid in methylene chloride provides a 96-member triamino-1,3,5-triazine library in 71-99% purities. A set of resin-bound triazines with chloro and benzylsulfonyl moieties are reacted with a set of 30 amines to compare the use of amino-substituted chlorotriazines, benzylthio-substituted chlorotriazines, and amino-substituted benzylsulfonyltriazines in substitution reactions with amines; substitution reactions of either amino-substituted sulfonyltriazines or benzylthio-substituted chlorotriazines gave the aminotriazine products in higher purities than reactions of amines with amino-substituted chlorotriazines.

ACCESSION NUMBER: 2003:148 CAPLUS
DOCUMENT NUMBER: 138:205020
TITLE: Novel Orthogonal Strategy toward Solid-Phase Synthesis of 1,3,5-Substituted Triazines
AUTHOR(S): Bork, Jacqueline T.; Lee, Jae Wook; Khersonsky, Sonya M.; Moon, Ho-Sang; Chang, Young-Tae
CORPORATE SOURCE: Department of Chemistry, New York University, New York, NY, 10003, USA
SOURCE: Organic Letters (2003), 5(2), 117-120
CODEN: ORLEF7; ISSN: 1523-7060
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 138:205020
REFERENCE COUNT: 17 THERE ARE 17 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 45 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB Heterocyclic β-amino acids are claimed for the prevention or treatment of epileptogenesis-associated diseases. Representative heterocyclic moieties are the following: thienyl, pyrrolyl, pyrimidyl, pyrazinyl, pyrazolyl, oxazolyl, isooxazolyl, thiazolyl, isothiazolyl, imidazolyl, furanyl, benzothiazolonyl, indolonyl, benzooxoazolinyl, benzothiophenyl, benzofuranyl, quinolinyl, isoquinolinyl, benzodioxazolyl, benzoxazolyl, benzothiazolyl, benzimidazolyl, methylenedioxyphenyl, ethylenedioxyphenyl, indolyl, purinyl, and deazapurinyl. Thus, 3-amino-3-(benzo[d]-1,3-dioxolan-5-yl)propionic acid was prepared by condensation of benzo[d]-1,3-dioxolane-5-carboxaldehyde with malonic acid and ammonium acetate.

ACCESSION NUMBER: 2002:927248 CAPLUS
DOCUMENT NUMBER: 138:4513
TITLE: Preparation of heterocyclic β-amino acids as antiepileptogenic agents
INVENTOR(S): Campbell, Allyson J.; Weaver, Donald F.
PATENT ASSIGNEE(S): Queen's University At Kingston, Can.
SOURCE: PCT Int. Appl., 75 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 2
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2002096424	A1	20021205	WO 2002-CA773	20020527
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM				
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				
EP 1397136	A1	20040317	EP 2002-729719	20020527
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR				
JP 2004536071	T2	20041202	JP 2002-592934	20020527
US 2003114441	A1	20030619	US 2002-222141	20020816
PRIORITY APPLN. INFO.:			US 2001-293495P P	20010525
			WO 2002-CA773 W	20020527

OTHER SOURCE(S): MARPAT 138:4513
REFERENCE COUNT: 8 THERE ARE 8 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 46 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB The composition contains addition-polymerizable unsatd. compound, a photoradical generator (e.g., organoboron compound), and R1NR2R3 [R1, R2 = H, (un)substituted aliphatic group; R3 = (un)substituted benzyl]. The composition shows improved sensitivity, storage stability, and fixability and is useful for a heat- and light-sensitive recording material or pressure- and light-sensitive recording material.

ACCESSION NUMBER: 2002:807319 CAPLUS
DOCUMENT NUMBER: 137:302204
TITLE: Photopolymerizable composition containing radical generator and amine, and recording material using it
INVENTOR(S): Matsumoto, Hirotaka; Washisu, Shintaro
PATENT ASSIGNEE(S): Fuji Photo Film Co., Ltd., Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 36 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2002308922	A2	20021023	JP 2001-114565	20010412
US 2003059705	A1	20030327	US 2002-120392	20020412
US 6869746	B2	20050322		
PRIORITY APPLN. INFO.:			JP 2001-114565 A	20010412

OTHER SOURCE(S): MARPAT 137:302204

L12 ANSWER 47 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB Diamide and amide-ester derivs. of imidazole-4,5-dicarboxylic acid form reliable H-bonding motifs in the solid state. The crystal structures of sym. substituted and dissym. substituted diamides as well as amide-ester combinations were analyzed to identify the intermol. H-bonding patterns. An intramol. seven-membered H-bonded conformation forms in all derivs. where the possibility existed due to the functionality present. The motifs observed for the diamides include intermol. NH···O and NH···N H-bonded dimers, with the exceptions to these motifs occurring in compds. having benzylamine substituents. The amines with a higher classification (i.e., 3° > 2° > 1°) in the dissym. substituted diamides are the intramol. H bond donors in the solid state, consistent with the capacity of the alkyl group to stabilize developing carbocation character resulting from bond polarization. The amide-ester derivs. also form an intramol. H bond and an intermol. motif based on NH···N and two different C2-H···O H bonds. A pyrrole amide-ester derivative forms an intramol. NH···O H bond in the solid state and an intermol. NH···O H-bonded chain. With the exception of the benzylamine-substituted diamides, the intermol. H-bonded motifs appear reliable for these imidazole-4,5-dicarboxylic acid derivs. and will be useful in the design of analogs for specific applications.

ACCESSION NUMBER: 2002:779161 CAPLUS
DOCUMENT NUMBER: 138:4321
TITLE: Intramolecular Hydrogen Bonding and Intermolecular Dimerization in the Crystal Structures of Imidazole-4,5-dicarboxylic Acid Derivatives
AUTHOR(S): Baures, Paul W.; Rush, Jeremy R.; Wiznycia, Alexander V.; Desper, John; Helfrich, Brian A.; Beatty, Alicia M.
CORPORATE SOURCE: Department of Chemistry, Kansas State University, Manhattan, KS, 66506, USA
SOURCE: Crystal Growth & Design (2002), 2(6), 653-664
CODEN: CGDEFU; ISSN: 1528-7483
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 138:4321
REFERENCE COUNT: 92 THERE ARE 92 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 48 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB A substantially quant. transfer of Cu(II) or Zn(II) salts from aqueous solution into a hydrocarbon (heptane or toluene) promptly occurs under CO2 in the presence of a dialkylamine (NHR2, R = Bu, CH2Ph). Recovery of the metal complexes from the organic phase affords Cu(O2CNR2)2(NHR2)2 or Zn4(μ4-O)(O2CNR2)6, resp., in high yield and purity. An x-ray diffraction study on a single crystal of Cu(O2CN(CH2Ph)2)2(NHBz2)2 1 showed the compound to be mononuclear with tetracoordinated Cu in an almost perfect square-planar geometry. The Zn derivative has the well-established oxo-centered tetranuclear structure (R = Bu, 2).

ACCESSION NUMBER: 2002:762840 CAPLUS
DOCUMENT NUMBER: 138:116808
TITLE: The NHR2/CO2 system as a metal ion extraction reagent from aqueous solution into hydrocarbons: copper(II) and zinc(II)
AUTHOR(S): Dell'Amico, Daniela Belli; Calderazzo, Fausto; Farnocchi, Saverio; Labella, Luca; Marchetti, Fabio
CORPORATE SOURCE: Dipartimento di Chimica e Chimica Industriale, Universita di Pisa, Pisa, I-56126, Italy
SOURCE: Inorganic Chemistry Communications (2002), 5(10), 848-852
CODEN: ICCOFP; ISSN: 1387-7003
PUBLISHER: Elsevier Science B.V.
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 138:116808
REFERENCE COUNT: 31 THERE ARE 31 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 49 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB Quant. thermodn. stability scales of organolithium compds. can be derived from measurements of Sn-Li exchange equilibrium A ΔGeq scale of α-oxy- and α-aminoorganolithium compds. was established, and quant. stabilization effects of O-alkyl, O-alkoxyalkyl, O-carbamoyl, N-carbamoyl, and O-carbonyl groups of the α-carbanion are presented. An α-oxycarbanion is far better stabilized by a carbonyl group as the O-substituent than by an alkyl or alkoxyalkyl group, while the anion-stabilizing effects of the different O-carbonyl substituents are comparable. An N-carbamoyl group has a somewhat higher stabilizing effect than its O-carbamoyl counterpart. NMR data are presented that show that benzylic N- or O-substituted carbanions have highly planarized structures where the neg. charge is highly delocalized. The stability data obtained from the Sn-Li exchanges can be easily converted into effective pK data that are useful for predicting the acid-base behavior of this type of organolithium species.

ACCESSION NUMBER: 2002:737848 CAPLUS
DOCUMENT NUMBER: 137:384887
TITLE: A Relative Organolithium Stability Scale Derived from Tin-Lithium Exchange Equilibria. Substituent Effects on the Stability of α-Oxy- and α-Aminoorganolithium Compounds
AUTHOR(S): Grana, Paula; Paleo, M. Rita; Sardina, F. Javier
CORPORATE SOURCE: Departamento de Quimica Organica Facultad de Quimica, Universidad de Santiago de Compostela, Santiago de Compostela, 15782, Spain
SOURCE: Journal of the American Chemical Society (2002), 124(42), 12511-12514
CODEN: JACSAT; ISSN: 0002-7863
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 137:384887
REFERENCE COUNT: 38 THERE ARE 38 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 50 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB Di-Et (S)-2,3-epoxypropylphosphonate [(S)-3] was transformed into (S)-phosphocarnitine [(S)-2] in the following sequence of reactions: a C-3 regioselective opening of the oxirane ring with magnesium bromide, quant. bromide displacement with trimethylamine, and ester hydrolysis. The epoxide ring opening of 3 with HCl/EtOAc gave a 92:8 mixture of 3- and 2-chloro-substituted phosphonates. Reaction of (S)-3 with aqueous NMe3 gave di-Et 3-hydroxy-1-propenylphosphonate as a major product.

ACCESSION NUMBER: 2002:646541 CAPLUS
DOCUMENT NUMBER: 138:24785
TITLE: An efficient synthesis of enantiomeric (S)-phosphocarnitine
AUTHOR(S): Wroblewski, Andrzej E.; Halajewska-Wosik, Anetta
CORPORATE SOURCE: Bioorganic Chemistry Laboratory, Faculty of Pharmacy, Medical University of Lodz, Lodz, 90-151, Pol.
SOURCE: European Journal of Organic Chemistry (2002), (16), 2758-2763
CODEN: EJOCFK; ISSN: 1434-193X
PUBLISHER: Wiley-VCH Verlag GmbH
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 138:24785
REFERENCE COUNT: 56 THERE ARE 56 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 51 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

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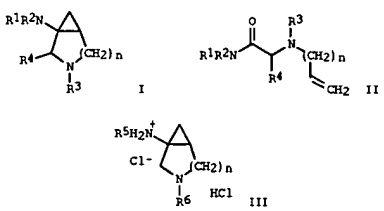

AB A variety of tris- and monoprotected derivs. with the 1-amino-3-azabicyclo[3.1.0]hexane and 1-amino-3-azabicyclo[4.1.0]heptane skeleton I (n = 1, 2; R1, R2 = Me, PhCH2; R3 = Me3COCO, PhCH2; R4 = H, Me3CSiMe2OCH2) were synthesized by intramol. reductive cyclopropanation of α-(N-allylamino)-substituted N,N-dialkylcarboxamides II. Starting from derivs. of the naturally occurring amino acid serine, the enantiomerically pure compds. I (n = 1; R1 = R2 = Me, PhCH2; R3 = PhCH2; R4 = Me3CSiMe2OCH2) were obtained with endo/exo ratios of 2-2.5:1 in 26-30% overall yields. X-ray crystal structure analyses of I (n = 1, 2; R1 = R2 = R3 = PhCH2; R4 = H) in each case found an equatorial position of the N-benzyl group on the heterocycle and a common boat conformation for the 3-azabicyclo[3.1.0]hexane and 3-azabicyclo[4.1.0]heptane skeletons as a whole. The unprotected bicyclic amine dihydrochlorides III (R5, R6 = H, Me) were prepared by palladium-catalyzed hydrogenative deprotection of I (R4 = H) under acidic conditions in 91-99% yields.

ACCESSION NUMBER: 2002:603570 CAPLUS
DOCUMENT NUMBER: 138:122509
TITLE: 3-Azabicyclo[3.1.0]hex-1-ylamines by Ti-mediated intramolecular reductive cyclopropanation of α-(N-allylamino)-substituted N,N-dialkylcarboxamides and carbonitriles
AUTHOR(S): Gensini, Martina; Kozhushkov, Sergei I.; Yufit, Dmitrii S.; Howard, Judith A. K.; Es-Sayed, Mazen; de Meijere, Armin
CORPORATE SOURCE: Institut fur Organische Chemie der Georg-August-Universitat Gottingen, Gottingen, 37077, Germany
SOURCE: European Journal of Organic Chemistry (2002), (15), 2499-2507
CODEN: EJOCFK; ISSN: 1434-193X
PUBLISHER: Wiley-VCH Verlag GmbH
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 138:122509
REFERENCE COUNT: 40 THERE ARE 40 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 52 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB A process for the conversion of gabapentin hydrochloride into gabapentin comprises dissoln. of gabapentin hydrochloride in a solvent in which the gabapentin hydrochloride and the gabapentin are completely soluble and subsequent addition of an amine that allows the removal of the chloride ion from the solution containing gabapentin hydrochloride; by precipitation of the hydrochloride of the same amine, leaving the gabapentin in solution in free amino acid form. This procedure using dicyclohexylamine afforded gabapentin in 80% yield and HPLC purity > 99.85% following treatment with Me and iso-Pr alcs.

ACCESSION NUMBER: 2002:428852 CAPLUS
DOCUMENT NUMBER: 136:401467
TITLE: A process for the preparation of 1-(aminomethyl)cyclohexaneacetic acid
INVENTOR(S): Ferrari, Massimo; Ghezzi, Marcello; Belotti, Paolo
PATENT ASSIGNEE(S): Erregierre S.P.A., Italy
SOURCE: PCT Int. Appl., 11 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2002044123	A1	20020606	WO 2001-EP13953	20011129
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM				
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				
IT 1319674	B1	20031023	IT 2000-MI2608	20001201
CA 2436908	AA	20020606	CA 2001-2436908	20011129
AU 2002029575	A5	20020611	AU 2002-29575	20011129
NZ 526370	A	20030829	NZ 2001-526370	20011129
EP 1347951	A1	20031001	EP 2001-990454	20011129
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR				
BR 2001015755	A	20031230	BR 2001-15755	20011129
JP 2004521875	T2	20040722	JP 2002-546493	20011129
ZA 2003004484	A	20040909	ZA 2003-4484	20030609
US 2005049432	A1	20050303	US 2003-433241	20031113

PRIORITY APPLN. INFO.: IT 2000-MI2608 A 20001201
WO 2001-EP13953 W 20011129
REFERENCE COUNT: 1 THERE ARE 1 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 53 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

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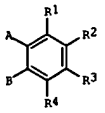


I

AB Title compds. are manufactured by reaction of trimellitamides I (R1-R4 = H, CONR5R6; ≥1 of R1-R3 = CONR5R6; A, B = CO2H, alkoxycarbonyl, carbamoyl, carboxylate, cyano; R5, R6 = Ph, benzyl, cyclohexyl), phthalic acid or its derivs. (except for I), urea, and Cu or its compds. followed by acid treatment. Thus, reaction of trimellitic anhydride diphenylamide, phthalic anhydride, urea, and CuCl gave blue products, which were treated with H2SO4 at room temperature for 4 h to give blue-purple pigment showing excellent stability after treatment with xylene under reflux.

ACCESSION NUMBER: 2002:421684 CAPLUS
DOCUMENT NUMBER: 136:403149
TITLE: Manufacture of solvent-stable α-copper phthalocyanines
INVENTOR(S): Endo, Atsushi; Kaneko, Tetsuya; Miyaji, Hidemitsu; Hondo, Hatsuo
PATENT ASSIGNEE(S): Toyo Ink Mfg. Co., Ltd., Japan; Kawasaki Kasei Chemicals, Ltd.
SOURCE: Jpn. Kokai Tokkyo Koho, 4 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2002161219	A2	20020604	JP 2000-360765	20001128

PRIORITY APPLN. INFO.: JP 2000-360765 20001128
OTHER SOURCE(S): MARPAT 136:403149

L12 ANSWER 54 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB Diastereomeric di-Et (1R,2R)- and (1S,2R)-2,3-epoxy-1-benzyloxypropylphosphonates were obtained from the resp. 2,3-O-cyclohexylidene-1-hydroxypropylphosphonates via the following sequence of reactions: benzylation, acetal hydrolysis and transformation of the terminal diols (1R,2R)- and (1S,2R)-(EtO)2P(O)CH(OCH2Ph)CH(OH)CH2OH thus obtained into epoxides using the Sharpless protocol. These epoxides were regioselectively opened with dibenzylamine to afford the title compds. (1R,2R)- and (1S,2R)-(EtO)2P(O)CH(OH)CH(OH)CH2NHAc after acetylation and hydrogenolysis.

ACCESSION NUMBER: 2002:403133 CAPLUS
DOCUMENT NUMBER: 137:247743
TITLE: Synthesis of diethyl (1R,2R)- and (1S,2R)-3-acetamido-1,2-dihydroxypropylphosphonates
AUTHOR(S): Wroblewski, Andrzej E.; Balcerzak, Katarzyna B.
CORPORATE SOURCE: Faculty of Pharmacy, Bioorganic Chemistry Laboratory, Medical University of Lodz, Lodz, 90-151, Pol.
SOURCE: Tetrahedron: Asymmetry (2002), 13(8), 845-850
CODEN: TASYE3; ISSN: 0957-4166
PUBLISHER: Elsevier Science Ltd.
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 137:247743
REFERENCE COUNT: 26 THERE ARE 26 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 55 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB Thirty-nine secondary amines were systematically investigated as additives in concentrated emeraldine base (EB)/NMP solns. for gelation and degradation When both the width (defined as the longest distance between 2 hydrogens in the plane perpendicular to the NH bond of the amine) and depth (defined as the longest distance between 2 atoms in a plane perpendicular to the width) of the amines are <4.53 Å and their pKa is >7.7, the amines significantly extend the gelation times of 20 mass % EB/NMP solns. for more than 12 h. However, some of these amines also significantly degrade the polymer. Amines with small width and depth and strong basicity, such as azetidine and pyrrolidine, can significantly destroy the EB structures. This was evidenced by order-of-magnitude decreases in doped film conductivity, by significantly changed UV-vis spectra, and by significantly reduced mol. wts. of the aged EB solns. as measured by gel permeation chromatog. (GPC). However, when both the width and depth of amines are >4.53 Å, these amines neither prolong gelation time nor appreciably degrade EB.

ACCESSION NUMBER: 2002:357930 CAPLUS
DOCUMENT NUMBER: 137:79635
TITLE: Physical Stabilization or Chemical Degradation of Concentrated Solutions of Polyaniline Emeraldine Base Containing Secondary Amine Additives
AUTHOR(S): Yang, Dali; Zuccarello, Guido; Mattes, Benjamin R.
CORPORATE SOURCE: Santa Fe Science and Technology Inc., Santa Fe, NM, 87505, USA
SOURCE: Macromolecules (2002), 35(13), 5304-5313
CODEN: MAMOEX; ISSN: 0024-9297
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
REFERENCE COUNT: 44 THERE ARE 44 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 56 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB With the purpose of developing a method of preparing Z-α,β-unsatd. amides, the Peterson reaction of the (triphenylsilyl)acetamide Ph3SiCH2COX (I; X = NBn2, NMe2) with various aldehydes was examined The reaction of aromatic aldehydes gave selectivities up to >97:3. It was found that the selectivity was a function of the electronic nature of the aromatic ring and higher Z selectivity was attained with electron-rich aldehydes. With aliphatic aldehydes selectivities up to 92:8 were achieved, and unlike with analogous phosphorus reagents, less sterically hindered aldehydes gave higher Z selectivity. Also, I (X = NMe2), which has a smaller amide group than I (X = NBn2), tended to give rise to higher selectivity. A comparison with the reaction of trimethylsilyl analogs revealed the significance of the Ph substituents on the silyl group.

ACCESSION NUMBER: 2002:348363 CAPLUS
DOCUMENT NUMBER: 137:78538
TITLE: Z-Selective Synthesis of α,β-Unsaturated Amides with Triphenylsilylacetamides
AUTHOR(S): Kojima, Satoshi; Inai, Hiroki; Hidaka, Tsugihiko; Fukuzaki, Tomohide; Ohkata, Katsuo
CORPORATE SOURCE: Department of Chemistry, Graduate School of Science, Hiroshima University, Kagamiyama Higashi-Hiroshima, 739-8526, Japan
SOURCE: Journal of Organic Chemistry (2002), 67(12), 4093-4099
CODEN: JOCEAH; ISSN: 0022-3263
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 137:78538
REFERENCE COUNT: 33 THERE ARE 33 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 57 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB A new "chemical tagging" method for homogeneous electrophilic scavenging is described. The method utilizes 5-norbornene-2-methanol to scavenge/tag a variety of electrophiles (p-toluenesulfonyl isocyanate, Ph isocyanate, or benzoyl chloride) that are present in excess. Once tagging is complete, the crude reaction mixture is subjected to a rapid (ring-opening metathesis polymerization) ROMP event utilizing the second generation Grubbs catalyst. This process yields a polymer that can be precipitated with methanol or ether/hexane, leaving products in excellent yield and purity.

ACCESSION NUMBER: 2002:315583 CAPLUS
DOCUMENT NUMBER: 137:64116
TITLE: Scavenge-ROMP-Filter: A Facile Strategy for Soluble Scavenging via Norbornenyl Tagging of Electrophilic Reagents
AUTHOR(S): Moore, Joel D.; Harned, Andrew M.; Henle, Julia; Flynn, Daniel L.; Hanson, Paul R.
CORPORATE SOURCE: Department of Chemistry, University of Kansas, Lawrence, KS, 66045-7582, USA
SOURCE: Organic Letters (2002), 4(11), 1847-1849
CODEN: ORLEF7; ISSN: 1523-7060
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
REFERENCE COUNT: 38 THERE ARE 38 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 58 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB α-Sulfinyl ketimines and β-sulfinyl enamines undergo reaction with delivery cyanide reagents such as (trimethylsilyl)cyanide or (tert-butyldimethylsilyl)cyanide in the presence of either stoichiometric excesses of ZnCl2 or ZnBr2, or catalytic amount of Yb(TfO)3. Ketimines included (-)-4-methoxy-N-[2-[(R)-(4-methylphenyl)sulfinyl]-1-phenylethylidene]benzenamine, (+)-3-[[(R)-(4-methylphenyl)sulfinyl]methyl]-1-oxa-4-azaspiro[4.5]dec-3-ene and (-)-N-[(1E)-2-[(R)-(4-methylphenyl)sulfinyl]ethenyl]-N-(phenylmethyl)benzenemethanamine. The use of ZnCl2 in alc. solvents provides the best diastereoselectivity., It is mediated by a chelated transition state, the p-tolyl group driving the anti attack of the reagent. By using Yb(TfO)3 poor diastereoselectivities but good yields are obtained. It seems that an iminium derivative originated by metal coordination with either the nitrogen or oxygen atom in the substrate is responsible for the observed results. Interestingly, β-sulfinyl enamines provide analogous α-amino nitriles in the same reaction conditions. It allowed the cyanosilylation of the covalently stabilized enamines arising from unstable β-sulfinyl aldehydes.

ACCESSION NUMBER: 2002:264520 CAPLUS
DOCUMENT NUMBER: 137:278955
TITLE: Stereoselective cyanosilylation of α-sulfinyl ketimines or its covalently stabilized enamine tautomers. Synthesis of enantiomerically pure α-sulfinylmethyl-α-amino nitriles
AUTHOR(S): Acherki, Hassan; Alvarez-Ibarra, Carlos; De Dios, Alfonso; Quiroga, Maria L.
CORPORATE SOURCE: Departamento de Quimica Organica, Facultad de Ciencias Quimicas, Ciudad Universitaria, Universidad Complutense, Madrid, 28040, Spain
SOURCE: Tetrahedron (2002), 58(16), 3217-3227
CODEN: TETRAB; ISSN: 0040-4020
PUBLISHER: Elsevier Science Ltd.
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 137:278955
REFERENCE COUNT: 63 THERE ARE 63 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 59 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB Anionic polymerization initiators useful in the preparation of polymers having a protected amine functional group are disclosed. The amine functionality includes a first protecting group, which can be aralkyl, Me, allyl or tertiary alkyl group. The other of the amine protecting groups can be the same as the first protecting group. Alternatively, the second protecting group can be different from the first protecting group, in which case it is selected to have differential stability to agents used to remove the aralkyl, Me, allyl or tertiary alkyl protecting group. 3-[(N-Benzyl-N-methyl)amino]-1-propyllithium was prepared and used in polymerization of isoprene.

ACCESSION NUMBER: 2002:240840 CAPLUS
DOCUMENT NUMBER: 136:279853
TITLE: Protected amino-functionalized anionic polymerization initiators and methods of making and using same
INVENTOR(S): Brockmann, Thorsten Werner; Hall, Randy W.
PATENT ASSIGNEE(S): FMC Corporation, USA
SOURCE: PCT Int. Appl., 92 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 3
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2002024764	A1	20020328	WO 2001-US22911	20010719
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM				
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				
US 6610859	B1	20030826	US 2000-665528	20000919
AU 2001080655	A5	20020402	AU 2001-80655	20010719
GB 2382076	A1	20030521	GB 2003-3022	20010719
DE 10196639	T	20030807	DE 2001-10196639	20010719
JP 2004513087	T2	20040430	JP 2002-529172	20010719
PRIORITY APPLN. INFO.:			US 2000-665528 A	20000919
			US 1999-256737 A2	19990224
			WO 2001-US22911 W	20010719

OTHER SOURCE(S): MARPAT 136:279853
REFERENCE COUNT: 14 THERE ARE 14 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 60 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB Reaction of nitrones with terminal alkynes takes place readily in the presence of a substoichiometric amount of diethylzinc in toluene, affording N-propargyl-hydroxylamines in excellent yields and purity.

ACCESSION NUMBER: 2002:234130 CAPLUS
DOCUMENT NUMBER: 136:385899
TITLE: Dialkylzinc-Assisted Alkynylation of Nitrones
AUTHOR(S): Pinet, Sandra; Pandya, Shashi Urvish; Chavant, Pierre Yves; Ayling, Alexander; Vallee, Yannick
CORPORATE SOURCE: LEDSS, UMR 5616, Universite J.Fourier, Grenoble, F-38041, Fr.
SOURCE: Organic Letters (2002), 4(9), 1463-1466
CODEN: ORLEF7; ISSN: 1523-7060
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 136:385899
REFERENCE COUNT: 55 THERE ARE 55 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 61 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB A simple colorimetric assay of various transition-metal catalysts showed that the combination of DPPF, Ni(COD)2, and acid is a highly active catalyst system for the hydroamination of dienes by alkylamines to form allylic amines. The scope of the reaction is broad; various primary and secondary alkylamines react with 1,3-dienes in the presence of these catalysts. Detailed mechanistic studies revealed the individual steps involved in the catalytic process. These studies uncovered unexpected thermodn. for the addition of amines to π-allyl nickel complexes: instead of the thermodn. favoring the reaction of a nickel allyl with an amine to form an allylic amine, the thermodn. favored reaction of a nickel(0) complex with allylic amine in the presence of acid to form a Ni(II) allyl. The realization of these thermodn. led us to the discovery that nickel and some palladium complexes in the presence or absence of acid catalyze the exchange of the amino groups of allylic amines with free amines. This exchange process was used to reveal the relative thermodn. stabilities of various allylic amines. In addition, this exchange reaction leads to racemization of allylic amines. Therefore, the relative rate for C-N bond formation and cleavage influences the enantioselectivity of diene hydroaminations.

ACCESSION NUMBER: 2002:198508 CAPLUS
DOCUMENT NUMBER: 136:354930
TITLE: A General Nickel-Catalyzed Hydroamination of 1,3-Dienes by Alkylamines: Catalyst Selection, Scope, and Mechanism
AUTHOR(S): Pawlas, Jan; Nakao, Yoshiaki; Kawatsura, Motoi; Hartwig, John F.
CORPORATE SOURCE: Department of Chemistry, Yale University, New Haven, CT, 06520-8107, USA
SOURCE: Journal of the American Chemical Society (2002), 124(14), 3669-3679
CODEN: JACSAT; ISSN: 0002-7863
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 136:354930
REFERENCE COUNT: 49 THERE ARE 49 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 62 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB The purple-red cesium 2-aza-allyl compound [{Cs(THF)}{N(CHPh)2}] (1) was obtained by the reaction of Cs in THF with HN(CH2Ph)2 with evolution of H2. 1 Was characterized by NMR, IR, and Raman spectra as well as by x-ray crystallog. In the solid state 1 forms infinite layers of {Cs(THF)}+ and [N(CHPh)2]- ions connected mainly by Cs+-π-electron interactions in the solid state. The layers are stacked along [001].

ACCESSION NUMBER: 2002:168108 CAPLUS
DOCUMENT NUMBER: 136:355261
TITLE: Direct Synthesis of a Cesium Azaallyl Compound
AUTHOR(S): Pauls, Jochen; Chitsaz, Soheila; Neumueller, Bernhard
CORPORATE SOURCE: Fachbereich Chemie, Universitaet Marburg, Marburg, D-35032, Germany
SOURCE: Organometallics (2002), 21(7), 1515-1517
CODEN: ORGND7; ISSN: 0276-7333
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 136:355261
REFERENCE COUNT: 23 THERE ARE 23 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 63 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB The invention provides an improved, tablet form for polymeric supports, which are used in organic synthesis in solvent media. More specifically, a fixed weight amount of beads of a functionalized polymer, which polymer is insol. in the reaction solvent for the intended synthesis, is provided as compressed tablets of essentially equal weight and composition The polymer beads are essentially intact, and are released as such when the tablets are disintegrated in the synthesis solvent. The invention tablets are characterized by the fact that they contain 0-20 weight% polyethylene glycol.

The tablets may also contain an addnl. non-functionalized polymer, such as polystyrene or PEG di-Me ether, as a disintegrating agent. This tablet form is useful in conventional synthesis, parallel synthesis, split-and-mix synthesis, and/or combinatorial chemical In a method for producing the tablets, beads of the functionalized polymer are compressed into tablets after pre-treatment with an aprotic organic solvent. For instance, one of 14 tablet compns. contained a 9:1 mixture of isocyanatomethyl polystyrene (1% divinylbenzene crosslinker) with PEG di-Me ether (mol. weight approx. 2000 Da). The tablets were 100 mg, with diameter 6 mm, and had a crushing strength of 16 N. They disintegrated rapidly (< 3 min) in CH2Cl2, THF, DMF, PhMe, MeCN, and DMSO, but were undisintegrated after 1 day in EtOH. The resulting dispersions were filterable, and the polymer beads undamaged as determined by SEM. In a performance test for attachment of organic amines to 4-[(4-nitrophenoxy)carbonyloxymethyl]phenoxymethyl polystyrene, the invention tablets gave increased yield and purity of product in 7 of 8 cases. For instance, in the case of 1-benzylpiperidin-4-ylamine, yield was increased from 62% to 90%, and purity (determined by UV) from 70 to 75%.

ACCESSION NUMBER: 2001:693276 CAPLUS
DOCUMENT NUMBER: 135:256832
TITLE: Tablet dosing form for a polymer support, use of said dosing form in organic chemical synthesis, and method for production of said dosing form
INVENTOR(S): Ruhland, Thomas; Holm, Per; Schultz, Kirsten; Egeskov Holm, Jannie; Andersen, Kim
PATENT ASSIGNEE(S): H. Lundbeck A/S, Den.
SOURCE: PCT Int. Appl., 30 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2001068598	A2	20010920	WO 2001-DK184	20010316
WO 2001068598	A3	20020221		
W: AE, AG, AL, AM, AT, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, CZ, DE, DE, DK, DK, DM, DZ, EE, EE, ES, FI, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM				
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG				
CA 2402584	AA	20010920	CA 2001-2402584	20010316

L12 ANSWER 63 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)

AU 2001044084	A5	20010924	AU 2001-44084		20010316
EP 1268050	A2	20030102	EP 2001-916930		20010316
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR					
JP 2003527372	T2	20030916	JP 2001-567694		20010316
US 2003138847	A1	20030724	US 2002-245839		20020916
PRIORITY APPLN. INFO.:			DK 2000-450	A	20000317
			WO 2001-DK184	W	20010316

OTHER SOURCE(S): CASREACT 135:256832

L12 ANSWER 64 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB A ketoester resin was developed as the basis for a selective scavenger for primary amines in the presence of secondary amines. The utility of the scavenger was demonstrated with a range of reductive amination chemistries with both mono- and diamines. Thus, R1COR2 (R1 = Ph, R2 = H; R1 = Pr, R2 = Me) reacted with R3 NHR3 (R3 = 2-furylmethyl, Ph2CH, 2-pyridylmethyl, etc.) to give R1R2CHNHR3. Treating the secondary amine product with the ketoester resin selectively removed the primary amine to give high purities and good yields of the secondary amine. The resin's specificity is based on the removal of the primary amines via their enamines.

ACCESSION NUMBER: 2001:572504 CAPLUS
DOCUMENT NUMBER: 136:69620
TITLE: Ketoester methacrylate resin, secondary amine clean-up in the presence of primary amines
AUTHOR(S): Yu, Zhanru; Alesso, Sonia; Pears, David; Worthington, Paul A.; Luke, Richard W. A.; Bradley, Mark
CORPORATE SOURCE: Department of Chemistry, University of Southampton, Southampton, SO17 1BJ, UK
SOURCE: Journal of the Chemical Society, Perkin Transactions 1 (2001), (16), 1947-1952
CODEN: JCSPCE; ISSN: 1472-7781
PUBLISHER: Royal Society of Chemistry
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 136:69620
REFERENCE COUNT: 18 THERE ARE 18 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

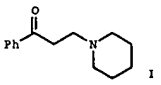
L12 ANSWER 65 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB Protected glycine analogs tethered to an imidazolidinone auxiliary undergo diastereoselective alkylation and acylation reactions in moderate to good yields (9-91%) with high levels of stereocontrol (generally >95% de). Subsequent alkylation of these derivs. has been demonstrated for the production of non-racemic α,α-disubstituted amino acid precursors. Diastereoselective aldol reactions are also found to proceed with good yields and excellent stereocontrol (62-84%, 93-95% de). Chiral auxiliary cleavage and hydrogenolysis of these adducts affords the β-hydroxy-α-amino acid derivs. with no observed erosion of optical purity.

ACCESSION NUMBER: 2001:537242 CAPLUS
DOCUMENT NUMBER: 135:289034
TITLE: Preparation of α-amino-carboxylic acid derivatives via diastereoselective reactions of glycine enolate equivalents
AUTHOR(S): Caddick, S.; Parr, N. J.; Pritchard, M. C.
CORPORATE SOURCE: School of Chemistry, Physics and Environmental Sciences, University of Sussex, Falmer, Brighton, BN1 9QJ, UK
SOURCE: Tetrahedron (2001), 57(30), 6615-6626
CODEN: TETRAB; ISSN: 0040-4020
PUBLISHER: Elsevier Science Ltd.
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 135:289034
REFERENCE COUNT: 51 THERE ARE 51 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 66 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

GI



I

AB A mild and efficient sequential transformation for the facile and rapid preparation of β-aminoketones or their derivs., e.g., pyrazolines, utilizing readily available and stable Weinreb amides as common starting materials is reported. The reaction proceeds in good to excellent yields for a variety of amides, vinyl Grignard reagents and N-nucleophiles. Thus, treating PhCONMe(OMe) with H2C:CHMgBr and piperidine gave β-aminoketone I in 95% yield.

ACCESSION NUMBER: 2001:294065 CAPLUS
DOCUMENT NUMBER: 135:121979
TITLE: Novel sequential process from N-methoxyamides and vinyl Grignard reagents: new synthesis of β-aminoketones
AUTHOR(S): Gomtsyan, Arthur; Koenig, Robert J.; Lee, Chih-Hung
CORPORATE SOURCE: Neurological and Urological Diseases Research, Abbott Laboratories, Abbott Park, IL, 60064, USA
SOURCE: Journal of Organic Chemistry (2001), 66(10), 3613-3616
CODEN: JOCEAH; ISSN: 0022-3263
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 135:121979
REFERENCE COUNT: 29 THERE ARE 29 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 67 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB The 1:1:1 complex of nitrosonium nitrate, 18-crown-6, and nitric acid [NO+•Crown•H(NO3-)2] acts as a efficient nitrosating agent for secondary alkyl and aryl amines to give N-nitrosamines in quant. yields. E.g., diethylamine, [NO+•Crown•H(NO3-)2] and silica are stirred in methylene chloride at ambient temperature for 5 min.; after rinsing the products through a plug of silica gel, N-nitroso-N,N-diethylamine is isolated in quant. yield. [NO+•Crown•H(NO3-)2] is prepared in quant. yield by bubbling a mixture of nitrogen dioxide and dinitrogen tetroxide through a solution of 18-crown-6 in methylene chloride followed by evaporation of solvent.
[NO+•Crown•H(NO3-)2] is an easily handled, stable, crystalline solid that rapidly nitrosates secondary amines under homogeneous conditions. N-nitrosoamines have been shown to be carcinogenic in laboratory animals and the products of N-nitrosation should thus be treated with caution.
ACCESSION NUMBER: 2001:268697 CAPLUS
DOCUMENT NUMBER: 135:60913
TITLE: N-Nitrosation of Secondary Amines with [NO+·Crown·H(NO3)2-]
AUTHOR(S): Zolfigol, Mohammad Ali; Zebarjadian, Mohammad Hassan; Chehardoli, Gholamabbas; Keypour, Hassan; Salehzadeh, Sadegh; Shamsipur, Mojtaba
CORPORATE SOURCE: Chemistry Department College of Science, Bu-Ali Sina University, Hamadan, 65174, Iran
SOURCE: Journal of Organic Chemistry (2001), 66(10), 3619-3620
CODEN: JOCEAH; ISSN: 0022-3263
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 135:60913
REFERENCE COUNT: 45 THERE ARE 45 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 68 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB The synthesis of new purine derivs. designed to inhibit cell cycle regulating cyclin-dependent kinases (CDKs), is reported. These compds., related to olomoucine and roscovitine, are characterized by the presence of a pyrrolidine methanol substituent at C-2 and a variety of ortho, meta and/or para substituents on the C-6 arylamino group.
ACCESSION NUMBER: 2001:223238 CAPLUS
DOCUMENT NUMBER: 135:19488
TITLE: Synthesis of a new series of purine derivatives and their anti-cyclin-dependent kinase activities
AUTHOR(S): Legraverend, Michel; Ludwig, Odile; Leclerc, Sophie; Meijer, Laurent
CORPORATE SOURCE: UMR 176 CNRS, Institut Curie, Section de Recherche, Centre Universitaire, Orsay, 91405, Fr.
SOURCE: Journal of Heterocyclic Chemistry (2001), 38(1), 299-303
CODEN: JHTCAD; ISSN: 0022-152X
PUBLISHER: HeteroCorporation
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 135:19488
REFERENCE COUNT: 16 THERE ARE 16 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 69 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB The use of 1H NMR for determination of the composition of a mixture is discussed. The use of 1H NMR for determination of the difference between the positional isomers 2-bromoethylbenzene and 1-bromoethylbenzene is noted. The use of 1H NMR in the preparation of diamines related to N-(2-phenyl-2-methylamino)ethylpyrrolidine is also discussed.
ACCESSION NUMBER: 2001:148766 CAPLUS
DOCUMENT NUMBER: 134:366546
TITLE: What's in a mixture?
AUTHOR(S): O'Brien, Peter
CORPORATE SOURCE: Department of Chemistry, University of York, UK
SOURCE: Chemistry Review (Deddington, United Kingdom) (2001), 10(3), 24-27
CODEN: CEEVE3; ISSN: 0959-8464
PUBLISHER: Philip Allan
DOCUMENT TYPE: Journal
LANGUAGE: English

L12 ANSWER 70 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB The products of the reduction of dihalo(diorganoamino)boranes with LiAlH4 in toluene depend upon the steric requirement of the amino substituents. It shows that upon using different procedures to produce secondary-amino(dihydro)boranes the results depend critically from the solvent, the stoichiometry of the educts and the temperature applied beyond the sterical factors. However, certain procedures are preferably used to produce distinct moieties. Eight procedures (in part using different ratios of the educts) were applied and evaluated for their results. Mixts. of products were explored by NMR and MS. Pure compds. are characterized by NMR: 1H, 11B, 13C, MS and elemental analyses or high resolution MS. An x-ray structure anal. is presented for dimeric piperidinoborane.
ACCESSION NUMBER: 2001:94884 CAPLUS
DOCUMENT NUMBER: 134:295854
TITLE: Reduction of piperidino- and related sec. amino(dihalo)boranes with LiAlH4 in toluene and related reactions
AUTHOR(S): Maringgele, Walter; Noltemeyer, Mathias; Teichgraber, Jorg; Meller, Anton
CORPORATE SOURCE: Institute of Inorganic Chemistry, University of Gottingen, Gottingen, D-37077, Germany
SOURCE: Main Group Metal Chemistry (2000), 23(12), 735-760
CODEN: MGMCE8; ISSN: 0792-1241
PUBLISHER: Freund Publishing House Ltd.
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 134:295854

L12 ANSWER 71 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB The solid-phase synthesis of 2,4-diaminoquinazolines is presented. The chemical involves the sequential condensation of 2-aminobenzonitriles and amines starting from an acyl isothiocyanate resin via a traceless cleavage and cyclization. The α-1 antagonist prazosin was synthesized, as well as several other examples, in good yields and purity.

ACCESSION NUMBER: 2001:58784 CAPLUS
DOCUMENT NUMBER: 134:252311
TITLE: Traceless Solid-Phase Synthesis of 2,4-Diaminoquinazolines
AUTHOR(S): Wilson, Lawrence J.
CORPORATE SOURCE: Healthcare Research Center, Procter & Gamble Pharmaceuticals, Mason, OH, 45040, USA
SOURCE: Organic Letters (2001), 3(4), 585-588
CODEN: ORLEF7; ISSN: 1523-7060
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 134:252311
REFERENCE COUNT: 11 THERE ARE 11 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 72 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

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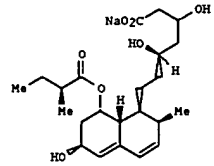
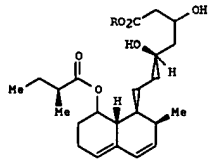


I



II

AB A process is provided for the bioconversion of compactin to pravastatin by a Micormonospora culture and the subsequent separation and purification of pravastatin. Specifically, the invention provides for the preparation of a pravastatin salt of formula I from a compactin salt of formula II where R+ represents an alkali metal or ammonium ion. In this process, microorganisms of the genera Micromonospora are aerobically cultivated in a suitable fermentation medium at 25-32 °C for a predetd. time at which a compactin salt is added and subsequently 6β-hydroxylated to form the corresponding pravastatin salt. The pravastatin salt formed during the fermentation may then be separated from the fermentation broth by adsorption on an anionic ion exchange resin, or by extraction with a water immiscible organic solvent followed by the the preparation of its lactone derivative or its secondary amine salt as an intermediate, or by purification of an aqueous alkaline extract obtained obtained from the organic solvent extract by liquid chromatog. on a non-ionic adsorbing resin. Thus, Micromonospora strain IDR-P3 was cultured for 72 h at 32 °C at which time 0.5 g/L sodium compactin was added to the fermentation broth which incubated for 72 h and which was followed by a second addition of 0.5 g/L of the compactin sodium salt followed by an addnl. 72 h incubation. After this second incubation, 75% of the compactin had been converted to the sodium salt of pravastatin. The fermentation broth was centrifuged, the supernatant was saved and the cell pellet was water washed. The supernatant and the wash were combined, the pH was adjusted to 3.5-4.0 with sulfuric acid and the pravastatin was

L12 ANSWER 72 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)

extd. with Et acetate. Then 150 mol% of dibenzyl amine was added to the ext. which was then concd. and held overnight at 0-5 °C. The pptd. pravastatin dibenzyl ammonium salt was recovered by filtration, and was ultimately purified ion exchange chromatog.

ACCESSION NUMBER: 2001:50841 CAPLUS
DOCUMENT NUMBER: 134:114919
TITLE: Microbial process for preparing pravastatin
INVENTOR(S): Jekkel, Antonia; Ambrus, Gabor; Ilkoy, Eva; Horvath, Ildiko; Konya, Attila; Szabo, Istvan Mihaly; Nagy, Zsuzsanna; Horvath, Gyula; Mozes, Julia; Barta, Istvan; Somogyi, Gyorgy; Salat, Janos; Boros, Sandor
PATENT ASSIGNEE(S): Gyogyszerkutato Intezet Kft., Hung.
SOURCE: PCT Int. Appl., 29 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 2
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2001004340	A1	20010118	WO 2000-HU66	20000629
W: AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM				
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG				
CA 2379015	AA	20010118	CA 2000-2379015	20000629
EP 1190087	A1	20020327	EP 2000-944121	20000629
EP 1190087	B1	20030618		
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO				
BR 2000013156	A	20020402	BR 2000-13156	20000629
TR 200200726	T2	20020621	TR 2002-200200726	20000629
NZ 516563	A	20021126	NZ 2000-516563	20000629
JP 2003504071	T2	20030204	JP 2001-509543	20000629
AT 243262	E	20030715	AT 2000-944121	20000629
EP 1327689	A1	20030716	EP 2003-75550	20000629
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, FI, RO, CY				
PT 1190087	T	20031031	PT 2000-944121	20000629
ES 2200891	T3	20040316	ES 2000-944121	20000629
AU 773633	B2	20040527	AU 2000-58355	20000629
RU 2235780	C2	20040910	RU 2002-103376	20000629
NO 2002000119	A	20020221	NO 2002-119	20020110
HR 2002000028	A1	20030630	HR 2002-28	20020110
ZA 2002000273	A	20030429	ZA 2002-273	20020111
BG 106302	A	20021031	BG 2002-106302	20020114

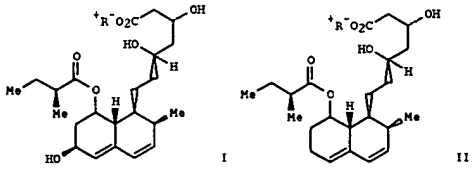
PRIORITY APPLN. INFO.:

HU 1999-2352	A	19990712
EP 2000-944121	A3	20000629
WO 2000-HU66	W	20000629

OTHER SOURCE(S): CASREACT 134:114919
REFERENCE COUNT: 2 THERE ARE 2 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 72 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)

L12 ANSWER 73 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
GI



I II

AB A process is provided for the bioconversion of compactin to pravastatin by a Micromonospora culture and the subsequent separation and purification of pravastatin. Specifically, the invention provides for the preparation of a pravastatin salt of formula I from a compactin salt of formula II where R+ represents an alkali metal or ammonium ion. In this process, microorganisms of the genera Micromonospora are aerobically cultivated in a suitable fermentation medium at 25-32 °C for a predetd. time at which a compactin salt is added and subsequently 6β-hydroxylated to form the corresponding pravastatin salt. The pravastatin salt formed during the fermentation may then be separated from the fermentation broth by adsorption on an anionic ion exchange resin, or by extraction with a water immiscible organic solvent followed by the the preparation of its lactone derivative or its secondary amine salt as an intermediate, or by purification of an aqueous alkaline extract obtained obtained from the organic solvent extract by liquid chromatog. on a non-ionic adsorbing resin. Thus, Micromonospora strain IDR-P3 was cultured for 72 h at 32 °C at which time 0.5 g/L sodium compactin was added to the fermentation broth which incubated for 72 h and which was followed by a second addition of 0.5 g/L of the compactin sodium salt followed by an addnl. 72 h incubation. After this second incubation, 75% of the compactin had been converted to the sodium salt of pravastatin. The fermentation broth was centrifuged, the supernatant was saved and the cell pellet was water washed. The supernatant and the wash were combined, the pH was adjusted to 3.5-4.0 with sulfuric acid and the pravastatin was extracted with Et acetate. Then 150 mol% of dibenzyl amine was added to the extract which was then concentrated and held overnight at 0-5 °C. The precipitated pravastatin dibenzyl ammonium salt was recovered by filtration, and was ultimately purified ion exchange chromatog.

ACCESSION NUMBER: 2001:50439 CAPLUS
DOCUMENT NUMBER: 134:114918
TITLE: Microbial process for preparing pravastatin
INVENTOR(S): Jekkel, Antonia; Ambrus, Gabor; Ilkoy, Eva; Horvath, Ildiko; Konya, Attila; Szabo, Istvan Mihaly; Nagy, Zsuzsanna; Horvath, Gyula; Mozes, Julianna; Barta, Istvan; Somogyi, Gyorgy; Salat, Janos; Boros, Sandor
PATENT ASSIGNEE(S): Ivax Corporation, USA
SOURCE: PCT Int. Appl., 31 pp.
CODEN: PIXXD2

L12 ANSWER 73 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 2
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2001003647	A2	20010118	WO 2000-US19384	20000711
WO 2001003647	A3	20010628		
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM				
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG				
TR 200200726	T2	20020621	TR 2002-200200726	20000629
EP 1327689	A1	20030716	EP 2003-75550	20000629
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, FI, RO, CY				
PT 1190087	T	20031031	PT 2000-944121	20000629
ES 2200891	T3	20040316	ES 2000-944121	20000629
CA 2373544	AA	20010118	CA 2000-2373544	20000711
AU 2000063492	A5	20010130	AU 2000-63492	20000711
EP 1198448	A2	20020424	EP 2000-950379	20000711
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL				
JP 2003528576	T2	20030930	JP 2001-508931	20000711
ZA 2002000273	A	20030429	ZA 2002-273	20020111

PRIORITY APPLN. INFO.: HU 1999-2352 A 19990712
EP 2000-944121 A3 20000629
WO 2000-US19384 W 20000711

OTHER SOURCE(S): CASREACT 134:114918

L12 ANSWER 74 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB Schiff bases were synthesized by addition of aldehyde or ketone followed by addition of benzyl azide to a solution of (PhCH2NEt3)2MoS4 in acetonitrile at room temperature All the Schiff bases were reduced to the arylamines. Dibenzylamine was produced by the reduction of the Schiff base obtained by the reduction of benzyl azide with (PhCH2NEt3)2MoS4 in acetonitrile. Dibenzylamine was further converted to its acylated derivative Reaction of (PhCH2NEt3)2MoS4 in acetonitrile with benzyl chloride produced dibenzyl disulfide in high yield and purity.

ACCESSION NUMBER: 2001:13723 CAPLUS
DOCUMENT NUMBER: 134:310935
TITLE: Synthesis based on benzyl chloride mediated by benzyltriethylammonium tetrathiomolybdate (PhCH2NEt3)2MoS4)
AUTHOR(S): Saha, Manoranjan; Chandrasekaran, S.
CORPORATE SOURCE: Department of Applied Chemistry and Chemical Technology, University of Dhaka, Dhaka, 1000, Bangladesh
SOURCE: Bangladesh Journal of Scientific and Industrial Research (1999), 34(1), 120-123
CODEN: BJSIBL; ISSN: 0304-9809
PUBLISHER: Bangladesh Council of Scientific and Industrial Research
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 134:310935
REFERENCE COUNT: 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 75 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB The use of the multi-component boronic Mannich reaction (BMR) in a solid-phase approach, in which an aryl boronic acid is combined with an aldehyde and a secondary amine is reported. Several examples are reported in which each of the three components is alternately anchored onto Wang polystyrene, giving in most cases (but not all) the expected products in high yields and purities. Based on 11B NMR studies, the intermediate formation of a tetracoordinated boron species could represent the prerequisite for success of the BMR is suggested.

ACCESSION NUMBER: 2000:854227 CAPLUS
DOCUMENT NUMBER: 134:207792
TITLE: The Boronic Mannich Reaction in a Solid-Phase Approach
AUTHOR(S): Schlienger, N.; Bryce, M. R.; Hansen, T. K.
CORPORATE SOURCE: Novo Nordisk A/S, Medicinal Chemistry Research IV, Maaloev, 2760, Den.
SOURCE: Tetrahedron (2000), 56(51), 10023-10030
CODEN: TETRAB; ISSN: 0040-4020
PUBLISHER: Elsevier Science Ltd.
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 134:207792
REFERENCE COUNT: 25 THERE ARE 25 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 76 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB Coupling of 2-chloro-5-aminobenzyl alc. to Merrifield resin (P-CH2Cl) and subsequent diazotization afforded polymer-bound diazonium ion [P]-CH2OCH2C6H3-2-Cl-5-N2+BF4- (3). DSC anal. of 3 and its 18-crown-6 and 21-crown-7 inclusion complexes indicated a high thermal stability, with decomposition significant at temps. higher than 90° and Ea for thermal decomposition of 114 kJ/mol (half-life for 3 of 11 h at 60° or 130 days at room temperature or 10 yr at 0°). Coupling of primary amines RNH2 with 3 gave the corresponding polymer-bound 1,3-disubstituted triazenes [P]-CH2OCH2C6H3-2-Cl-5-N:NNHR which underwent regioselective reactions at the N3 nitrogen of the triazene group and cleavage to give RNHR'. The use of 3 as a scavenger resin for removal of amines, anilines, and phenols was also discussed.

ACCESSION NUMBER: 2000:755914 CAPLUS
DOCUMENT NUMBER: 134:41779
TITLE: The first stable diazonium ion on solid support-investigations on stability and usage as linker and scavenger in solid-phase organic synthesis
AUTHOR(S): Dahmen, Stefan; Brase, Stefan
CORPORATE SOURCE: Institut fur Organische Chemie der Technischen Hochschule Aachen, Aachen, 52074, Germany
SOURCE: Angewandte Chemie, International Edition (2000), 39(20), 3681-3683
CODEN: ACIEF5; ISSN: 1433-7851
PUBLISHER: Wiley-VCH Verlag GmbH
DOCUMENT TYPE: Journal
LANGUAGE: English
REFERENCE COUNT: 17 THERE ARE 17 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 77 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB Several novel multidentate dinucleating ligands based on 1,8-naphthyridine have been synthesized in which the 1,8-naphthyridine moiety serves as a bridging unit. These ligands can link two metal ions like the syn, syn coordination mode of bridging carboxylate groups encountered in a variety of dimetallic centers in biol. Stable dimetallic complexes with variable metal-metal separations and geometries readily form with the use of these ligands.

ACCESSION NUMBER: 2000:720127 CAPLUS
DOCUMENT NUMBER: 134:56595
TITLE: Design and Synthesis of Multidentate Dinucleating Ligands Based on 1,8-Naphthyridine
AUTHOR(S): He, C.; Lippard, S. J.
CORPORATE SOURCE: Department of Chemistry, Massachusetts Institute of Technology, Cambridge, MA, 02139, USA
SOURCE: Tetrahedron (2000), 56(42), 8245-8252
CODEN: TETRAB; ISSN: 0040-4020
PUBLISHER: Elsevier Science Ltd.
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 134:56595
REFERENCE COUNT: 50 THERE ARE 50 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

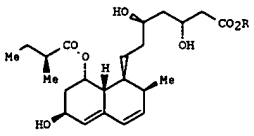
L12 ANSWER 78 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB The synthesis of 2-aminoimidazolinones from resin-bound amino acids is described. Reaction of resin-bound amino acids with isothiocyanates followed by treatment of the resulting thioureas with Mukaiyama's reagent afforded the corresponding carbodiimides, which reacted with amines to give 2-aminoimidazolinones in good yield and purity through a cyclization reaction that cleaves the product from the resin.

ACCESSION NUMBER: 2000:619113 CAPLUS
DOCUMENT NUMBER: 133:362728
TITLE: Solid-phase synthesis of 2-aminoimidazolinones
AUTHOR(S): Drewry, D. H.; Ghiron, C.
CORPORATE SOURCE: Combichem Technology Team, Glaxo Wellcome, Inc., Research Triangle Park, NC, 27709, USA
SOURCE: Tetrahedron Letters (2000), 41(36), 6989-6992
CODEN: TELEAY; ISSN: 0040-4039
PUBLISHER: Elsevier Science Ltd.
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 133:362728
REFERENCE COUNT: 11 THERE ARE 11 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 79 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

GI



I

AB A method for the preparation of pravastatin I (R = H) and its salts I (R = Na, dibenzylamine, dioctylamine, dicyclohexylamine, etc.) via fermentation of compactin using using the filamentous mold, Mortierella maculata, was described. Thus, bioconversion of compactin using Mortierella maculata in a medium of 50 g of glucose, 20 g of soybean meal, and 1000 mL water resulted in the formation of pravastatin. The pravastatin was purified via formation of its dibenzylamine salt. Novel strains of Mortierella maculata were also disclosed.

ACCESSION NUMBER: 2000:553532 CAPLUS
DOCUMENT NUMBER: 133:149265
TITLE: Preparation of pravastatin by fermentation using the filamentous mold, Mortierella maculata
INVENTOR(S): Jekkel, Antonia; Konya, Attila; Barta, Istvan; Ilkoy, Eva; Somogyi, Gyorgy; Ambrus, Gabor; Horvath, Gyula; Albrecht, Karoly; Szabo, Istvan M.; Mozes Suto, Julianna; Salat, Janos; Andor, Attila; Biricsik, Laszlo; Boros, Sandor; Lang, Ildiko; Bidlo Igloy, Margit
PATENT ASSIGNEE(S): Institute for Drug Research Ltd., Hung.; Teva Pharmaceuticals USA, Inc.
SOURCE: PCT Int. Appl., 42 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2000046175	A1	20000810	WO 2000-US2993	20000203
W: AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM				
RW: GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG				
CA 2361701	AA	20000810	CA 2000-2361701	20000203
AU 2000033567	A5	20000825	AU 2000-33567	20000203
AU 774438	B2	20040624		
EP 1154979	A1	20011121	EP 2000-911709	20000203

L12 ANSWER 79 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO

BR 2000009180	A	20020326	BR 2000-9180	20000203
TR 200103127	T2	20020422	TR 2001-200103127	20000203
JP 2002535977	T2	20021029	JP 2000-597248	20000203
US 6682913	B1	20040127	US 2000-497805	20000203
EP 1491522	A1	20041229	EP 2004-23144	20000203
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL				
HR 2001000577	A1	20021231	HR 2001-577	20010731
ZA 2001006359	A	20020802	ZA 2001-6359	20010802
NO 2001003818	A	20011003	NO 2001-3818	20010803
BG 105778	A	20020531	BG 2001-105778	20010803
US 2002081675	A1	20020627	US 2001-11176	20011205
US 6750366	B2	20040615		
US 2003207413	A1	20031106	US 2003-437058	20030514
US 6696599	B2	20040224		
US 2004039225	A1	20040226	US 2003-648386	20030827
JP 2005047924	A2	20050224	JP 2004-254575	20040901

PRIORITY APPLN. INFO.:

US 1999-118458P	P	19990203
US 1999-134759P	P	19990518
EP 2000-911709	A3	20000203
JP 2000-597248	A3	20000203
US 2000-497805	A3	20000203
WO 2000-US2993	W	20000203
US 2001-11176	A3	20011205

OTHER SOURCE(S): MARPAT 133:149265
REFERENCE COUNT: 1 THERE ARE 1 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 80 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB The first example of a fully automated solution-phase parallel synthesis method including online product purifn, AutoChem, is described. The versatile generic pipetting routines, user-friendly software, and simple organization by racks of common reagents, diversity reagents, and reaction vessels allow the chemist to perfrom different chemistries in a straightforward fashion. The preparation of 32 pure products from Borch redns. in one week exemplifies the utility of this method.
ACCESSION NUMBER: 2000:500169 CAPLUS
DOCUMENT NUMBER: 133:252086
TITLE: AutoChem: Automated Solution-Phase Parallel Synthesis and Purification via HPLC
AUTHOR(S): Tommasi, Ruben A.; Whaley, Louis W.; Marepalli, Hanumantha R.
CORPORATE SOURCE: Novartis Pharmaceuticals Corporation, Summit, NJ, 07901, USA
SOURCE: Journal of Combinatorial Chemistry (2000), 2(5), 447-449
CODEN: JCCHFF; ISSN: 1520-4766
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
REFERENCE COUNT: 17 THERE ARE 17 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 81 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB N-alkoxy(or aryloxy)carbonyl isothiocyanate derivs. R1O2CNHC(:S)YR4 [R1 = C1-8 alkyl, C2-4 alkenyl, C6-10 aryl; R4 = C1-10 alkyl, C6-10 aryl, C1-8 alkoxy; Y = O, S, NR5; R5 = H, R4] (e.g., N-methoxycarbonyl-O-Me thionocarbamate) are prepared by reacting a haloformate ester XCO2R1 (X = halogen) (e.g., Me chloroformate) with a thiocyanate MSCN (M = alkali metal, alkaline earth metal, NH4) (e.g., sodium thiocyanate) in the presence of an organic solvent (e.g., MIBK) and a catalytic amount of an N,N-dialkylarylamine (e.g., N,N-dimethylaniline) to produce an N-alkoxy(or aryloxy)carbonyl isothiocyanate intermediate S:C:NCO2R1 (e.g., N-methoxycarbonyl isothiocyanate) which then undergoes an addition reaction with an alc., mercaptan, or amine R4YH (e.g., methanol) to give the N-alkoxy(or aryloxy)carbonyl isothiocyanate derivative in high yield and purity.
ACCESSION NUMBER: 2000:344129 CAPLUS
DOCUMENT NUMBER: 132:321675
TITLE: Process for manufacturing N-alkoxy(or aryloxy)carbonyl isothiocyanate derivatives using N,N-dialkylarylamines as catalysts
INVENTOR(S): Kulkarni, Shekhar V.
PATENT ASSIGNEE(S): Bayer Corporation, USA
SOURCE: U.S., 5 pp.
CODEN: USXXAM
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 2
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 6066754	A	20000523	US 1999-329744	19990610
EP 1059289	A1	20001213	EP 2000-110990	20000529
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO				
CA 2310984	AA	20001210	CA 2000-2310984	20000605
BR 2000002599	A	20010102	BR 2000-2599	20000608
CN 1277190	A	20001220	CN 2000-118085	20000609
JP 2001026576	A2	20010130	JP 2000-173668	20000609

PRIORITY APPLN. INFO.:

US 1999-329405	A	19990610
US 1999-329744	A	19990610

OTHER SOURCE(S): CASREACT 132:321675; MARPAT 132:321675
REFERENCE COUNT: 5 THERE ARE 5 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 82 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB A combination of inorg. acidic salts or silica gel supported inorg. acids and sodium nitrite in the presence of wet SiO2 was used as an effective nitrosating agent for the nitrosation of secondary amines to their corresponding nitroso derivs. under mild and heterogeneous conditions in moderate to excellent yields. Mg(HSO4)2 and NaHSO4 are superior to all the aforementioned reagents in convenience, yield and purity of the isolated nitrosamines.
ACCESSION NUMBER: 2000:310891 CAPLUS
DOCUMENT NUMBER: 133:104617
TITLE: An efficient method for N-nitrosation of secondary amines under mild and heterogeneous conditions
AUTHOR(S): Zolfigol, Mohammad Ali; Ghaemi, Ezat; Madrakian, Elahe; Kiany-Borazjani, Maryam
CORPORATE SOURCE: Chemistry Department, College of Science, Bu-Ali Sina University, Hamadan, 65174, Iran
SOURCE: Synthetic Communications (2000), 30(11), 2057-2060
CODEN: SYNCAV; ISSN: 0039-7911
PUBLISHER: Marcel Dekker, Inc.
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 133:104617
REFERENCE COUNT: 4 THERE ARE 4 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 83 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB Two sample preparation methods for the determination of dibenzylamine (DBA) in artificial saliva leachates from rubber baby bottle nipples have been developed, using either solid-phase extraction (SPE) with N-vinylpyrrolidone/divinylbenzene as the sorbent or solid-phase microextn. (SPME) with a polyacrylate coated fiber. The baby bottle nipples were immersed into artificial saliva for 6 h, a part of the solution was brought to pH 9 for SPE or pH 10 for SPME and the analyte was extracted by SPE or SPME. After elution with Et acetate (SPE) or thermal desorption (SPME) DBA was determined by gas chromatog. with mass spectrometric detection. The main advantages of SPE were superior ruggedness and stability as well as the possibility of preparing several samples simultaneously. SPME offered a greater sensitivity and much smaller sample vols. were required. The results obtained for the investigated rubber baby bottle nipples were almost identical with both the methods showing deviations of less than 3%.

ACCESSION NUMBER: 2000:307309 CAPLUS
DOCUMENT NUMBER: 133:88372
TITLE: Direct comparison of solid-phase extraction and solid-phase microextraction for the gas chromatographic determination of dibenzylamine in artificial saliva leachates from baby bottle teats
AUTHOR(S): Niessner, G.; Klampfl, C. W.
CORPORATE SOURCE: Department of Analytical Chemistry, Johannes Kepler University Linz, Linz, A-4040, Austria
SOURCE: Analytica Chimica Acta (2000), 414(1-2), 133-140
CODEN: ACACAM; ISSN: 0003-2670
PUBLISHER: Elsevier Science B.V.
DOCUMENT TYPE: Journal
LANGUAGE: English
REFERENCE COUNT: 14 THERE ARE 14 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 84 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB Nucleophilic addition (Nu-M+) to isolevoglucosenone generates enolates stereospecifically (exo face addition) that can be reacted with sugar-derived aldehydes to give C(1→3)-linked disaccharide precursors with high diastereoselectivity. Limitations of the method arising from unfavorable aldolate stability can be overcome by using Et2AlI as the nucleophile. This leads to products of Baylis-Hillmann condensations. One example is presented and has led to the preparation of 2,3-anhydro-3-C-[(1S)-2,6-anhydro-D-glycero-D-gulo-heptitol-1-C-yl]-β-D-gulo-pyranose.

ACCESSION NUMBER: 2000:184009 CAPLUS
DOCUMENT NUMBER: 133:4869
TITLE: Convergent syntheses of C(1→3)-linked disaccharides starting from isolevoglucosenone
AUTHOR(S): Zhu, Yao-Hua; Demange, Raynald; Vogel, Pierre
CORPORATE SOURCE: Section de Chimie, BCH, l'Universite de Lausanne, Lausanne-Dorigny, CH-1015, Switz.
SOURCE: Tetrahedron: Asymmetry (2000), 11(1), 263-282
CODEN: TASYE3; ISSN: 0957-4166
PUBLISHER: Elsevier Science Ltd.
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 133:4869
REFERENCE COUNT: 84 THERE ARE 84 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

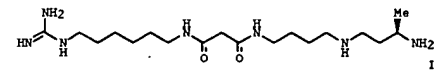
L12 ANSWER 85 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB The crystal structure of the prepared stable toluene solvate of bis(N1,N1,N5,N5-tetrabenzyl-2,4-dithiobiureto)nickel(II) shows that the solvent mols. are held within lattice cavities of well-defined size and shape. Recrystn. from a mixture of xylenes yields selectively the p-xylene solvate.

ACCESSION NUMBER: 1999:798762 CAPLUS
DOCUMENT NUMBER: 132:101875
TITLE: Shape selective solvent inclusion within the lattice of bis(N1,N1,N5,N5-tetrabenzyl-2,4-dithiobiureto)nickel(II)
AUTHOR(S): Billson, Timothy S.; Crane, Jonathan D.; Sinn, Ekkehard; Teat, Simon J.; Wheeler, Eleanor; Young, Nigel A.
CORPORATE SOURCE: Department of Chemistry, The University of Hull, Kingston-upon-Hull, HU6 7RX, UK
SOURCE: Inorganic Chemistry Communications (1999), 2(11), 527-529
CODEN: ICCOFP; ISSN: 1387-7003
PUBLISHER: Elsevier Science S.A.
DOCUMENT TYPE: Journal
LANGUAGE: English
REFERENCE COUNT: 17 THERE ARE 17 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 86 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

GI



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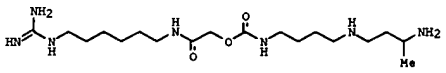

II

AB A series of new analogs of 15-deoxyspergualin (DSG), an immunosuppressive agent commercialized in Japan, was synthesized and tested in a graft-vs.-host disease (GVHD) model in mice. Various substitutions of the spermidine "D" region were made in order to determine its optimum structure in terms of in vivo immunosuppressive activity. Various positions of methylation were first investigated leading to the discovery of the monomethylated malonic derivative I in which the pro-R hydrogen of the methylene α to the primary amine of the spermidine moiety has been replaced by a Me group. Synthesis of the similarly methylated analog of the previously reported glycolic derivative LF 08-0299 afforded II which demonstrated a powerful activity at a dose as low as 0.3 mg/kg in the GVHD model and was much more potent than DSG in the demanding heart allotransplantation model in rats. The improvement of in vivo activity was supposed to be related to an increase of the metabolic stability of the methylated analogs compared to the parent mols. Due to its very low active dose, compatible with a s.c. administration in humans, and its favorable pharmacol. and toxicol. profile, II was selected as a candidate for clin. evaluation.

ACCESSION NUMBER: 1999:694705 CAPLUS
DOCUMENT NUMBER: 132:35536
TITLE: Structure-Immunosuppressive Activity Relationships of New Analogues of 15-Deoxyspergualin. 2. Structural Modifications of the Spermidine Moiety
AUTHOR(S): Lebreton, Luc; Jost, Eric; Carboni, Bertrand; Annat, Jocelyne; Vaultier, Michel; Dutartre, Patrick; Renaut, Patrice
CORPORATE SOURCE: Axe Immunologie, Daix, 21121, Fr.
SOURCE: Journal of Medicinal Chemistry (1999), 42(23), 4749-4763
CODEN: JMCMAR; ISSN: 0022-2623
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
REFERENCE COUNT: 28 THERE ARE 28 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 87 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
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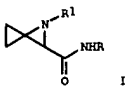

I

AB Azaspiropentanecarboxamides I [R = H with R1 = PhCH2, Ph(CH2)2; R, R1 = PhCH2; R, R1 = 4-MeOC6H4CH2; R = 4-MeOC6H4CH2, R1 = PhCH2; R = (S)-PhMeCH, R1 = PhCH2; R = PhCH2, R1 = (S)-PhMeCH] are formed with remarkable ease in 2 steps in a 1-pot operation from Me 2-chloro-2-cyclopropylideneacetate by addition of a primary amine in THF and subsequent treatment with NaH/Et3N in the presence of another equivalent of a primary amine or NH3. Achievable yields of I were moderate to good, while the corresponding esters could only be obtained in poor yields. The new α-amino amides are surprisingly stable and can be incorporated into small peptides as demonstrated with the preparation of a glycinyl peptide and a spirocyclopropaneoxazoline.

ACCESSION NUMBER: 1999:559531 CAPLUS
DOCUMENT NUMBER: 131:286791
TITLE: Cyclopropyl building blocks in organic synthesis. Part 51. An easy access to 1-azaspiropentane-2-carboxamides. The first derivatives of a new type of amino acids
AUTHOR(S): Tamm, Markus; Thutewohl, Michael; Ricker, Carsten B.; Bes, M. Teresa; De Meijere, Armin
CORPORATE SOURCE: Institut Organische Chemie, Georg-August-Univ., Gottingen, D-37077, Germany
SOURCE: European Journal of Organic Chemistry (1999), (9), 2017-2024
CODEN: EJOCFK; ISSN: 1434-193X
PUBLISHER: Wiley-VCH Verlag GmbH
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 131:286791
REFERENCE COUNT: 47 THERE ARE 47 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 88 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB This study explains and introduces novel catalyst systems, by fundamental studies, in all-water blown polyurethane (PUR) spray foam applications and CFC free polyisocyanurate (PIR) sprayed foam applications. The elimination of CFC in PUR applications has successfully been achieved in most cases and alternative blowing agents such as HCFC-141b, pentane, cyclopentane, water are commonly used today. For the spray application, HCFC-141b is the primary blowing agent, however, HCFC-141b will be phased out by the year 2003. Other alternative blowing agents have been investigated and water is also being considered as the good candidate. All-water blown systems however, have many problems, such as delay of the initial blow, foam cracking due to the high reaction exotherm, high d., adhesive strength and so on. The catalyst plays an important role to improve spray foam systems, and a wide selection of catalysts, such as tertiary amines catalysts and metal based catalysts have been proposed. Most catalysts, however, cannot meet recent manufs. requirements. For example, the use of blowing amine catalyst is effective in order to make the initial activity faster in general, however in all-water blown spray foam applications there is a limit for shortening the cream time even though increased concentration levels of conventional blowing
catalyst are utilized. In the case of using a high concentration level of blowing amine catalyst, the adhesive strength becomes poor due to the high content of urea linkages. Furthermore, a high concentration level of conventional blowing amine catalysts also contributes to high odor in the foam. TOSOH corporation has investigated the above areas from the standpoint of tertiary amine catalysts and has successfully developed the novel amine catalysts systems TOYOCAT-FB20 and FB30. In contrast to the conventional amine catalysts, TOYOCAT-FB20 and FB30 enables one to achieve fast initial blowing activity identical to HCFC-141b blown systems. It is also possible to prevent the "hanging" of the foam and to produce good foam efficiency such as low d. foam, good moldability and so on. TOYOCAT-FB20 and FB30 can improve the adhesive strength and reduce odor thereby improve the working environment. In case of PIR spray foam, the delay in initial blowing occurs at low temperature even when using HCFC-141b.
TOYOCAT FB20 and FB30 can be applied to PIR spray foam system and enables one to achieve desired fast initial blowing activity. Foam d. can also be reduced without sacrificing acceptable flammability. This technol. assists in the successful production of spray foam systems with excellent phys. properties, including fast initial blowing activity, improved moldability, friability and low d. foam.

ACCESSION NUMBER: 1999:496385 CAPLUS
DOCUMENT NUMBER: 132:123587
TITLE: The function of tertiary amine catalyst systems in sprayed foams
AUTHOR(S): Kometani, H.; Tamano, Y.; Ishida, M.; Lowe, D. W.
CORPORATE SOURCE: Chemical Research Laboratory, TOSOH Corporation, Yamaguchi, 746, Japan
SOURCE: Polyurethanes Expo '98, Proceedings, Dallas, Sept. 17-20, 1998 (1998), 239-246. Society of the Plastics Industry: Washington, D. C.
CODEN: 67XLAZ
DOCUMENT TYPE: Conference
LANGUAGE: English
REFERENCE COUNT: 6 THERE ARE 6 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 89 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB The reactions of triethylaluminum with 13 secondary amines (RH = HNMe2, HNEt2, HNPr2n, HNPr2i, HNBu2n, HNBu2i, HNBu2s, HNC4H8, HNC5H10, HNC6H12, HN(c-C6H11)2, HN(CH2Ph)2, and HNC4H8NMe) afford room-temperature stable , clear, colorless liquid complexes. These complexes were characterized by 1H and 13C NMR, IR and elemental analyses. Trends in the NMR chemical shift data are compared with data previously reported for the analogous trimethylaluminum, -gallium, and -indium compds. in terms of the steric properties of the amines. Subsequent thermolysis of these complexes yields dimeric aminoalanes via 1,2-elimination of ethane in all cases. The dimers were characterized by 1H and 13C NMR, IR, m.p., cryoscopic mol. weight detns., and elemental analyses. The NMR chemical shift data are compared
with known data for the [Me2AlR]2 and [Me2GaR]2 series. The mol. structures of [Et2AlN(c-C6H11)2]2 and [Et2AlNC4H8NCH3]2, obtained from x-ray crystal data, are presented and discussed in terms of the correlations between the structural parameters of the Al2N2 ring and the nature of the Al and N substituents.

ACCESSION NUMBER: 1999:404280 CAPLUS
DOCUMENT NUMBER: 131:130033
TITLE: Reactivity of triethylaluminum with a series of secondary amines. Adduct and aminoalane dimer synthesis and characterization; the crystal structures of [Et2AlN(c-C6H11)2]2 and [Et2AlNC4H8NCH3]2
AUTHOR(S): Styron, Eric K.; Lake, Charles H.; Schauer, Steven J.; Watkins, Charles L.; Krannich, Larry K.
CORPORATE SOURCE: Department of Chemistry, University of Alabama at Birmingham, Birmingham, AL, 35294-1240, USA
SOURCE: Polyhedron (1999), 18(11), 1595-1602
CODEN: PLYHDE; ISSN: 0277-5387
PUBLISHER: Elsevier Science Ltd.
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 131:130033
REFERENCE COUNT: 34 THERE ARE 34 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 90 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB Pentacyanonitrosylferrate(II) (I) reacts with n-butylamine to produce di-n-butylamine in high yields (81-95%). The absence of rearranged products indicates that the initially produced diazonium ion is stabilized by coordination to the metal. Benzylamine and 1,4-diaminobutane react with I to produce dibenzylamine and piperidine, resp.

ACCESSION NUMBER: 1999:380233 CAPLUS
DOCUMENT NUMBER: 131:129568
TITLE: The reaction of pentacyanonitrosylferrate(II) with primary amines as a source of stabilized aliphatic diazonium ions: a new route to secondary amines
AUTHOR(S): Doctorovich, Fabio; Trapani, Cecilia
CORPORATE SOURCE: Departamento de Quimica Inorganica, Analitica y Quimica Fisica/INQUIMAE, Facultad de Ciencias Exactas y Naturales, Universidad de Buenos Aires, Buenos Aires, 1428, Argent.
SOURCE: Tetrahedron Letters (1999), 40(25), 4635-4638
CODEN: TELEAY; ISSN: 0040-4039
PUBLISHER: Elsevier Science Ltd.
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 131:129568
REFERENCE COUNT: 16 THERE ARE 16 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 91 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB This paper describes the successful transfer of benzotriazole-based chemical on solid support. The strategy followed to anchor this peculiar heterocycle on solid phase and the full anal. characterization of the various supported benzotriazoles are herein described. The chemical assessment process on solid phase, the preparation of discrete libraries by parallel synthesis, the semiautomated purification procedures, and the complete anal. characterization of the library components are also presented and discussed.

ACCESSION NUMBER: 1999:361140 CAPLUS
DOCUMENT NUMBER: 131:184908
TITLE: Solid-Supported Benzotriazoles: Synthetic Auxiliaries and Traceless Linkers for the Combinatorial Synthesis of Amine Libraries
AUTHOR(S): Paio, Alfredo; Zaramella, Alessio; Ferritto, Rafael; Conti, Nadia; Marchioro, Carla; Seneci, Pierfausto
CORPORATE SOURCE: GlaxoWellcome Medicines Research Centre, Verona, 37135, Italy
SOURCE: Journal of Combinatorial Chemistry (1999), 1(4), 317-325
CODEN: JCCHFF; ISSN: 1520-4766
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 131:184908
REFERENCE COUNT: 53 THERE ARE 53 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 92 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB Starting from closo-[B10H10]2- hydrophobic monoanions [R1R2R3N-B10H9]- (R = H, PhCH2, Ph, Me, dimethyloctyl) could be obtained by a multistep process in which the displacement of N from [1-NZB10H9]- by amines was the key step. Attempts at direct synthesis employing bulky tertiary amines were unsuccessful: no reaction occurred at 120° and at 150° [1-N2B10H9]- decomposed to [B20H18]2-. Pd(PPh3)2Cl2 used as a catalyst produced a favorable effect, but the [R1R2R3N-B10H9]- ions were present in too low concentration to be isolated from the reaction mixts. A more suitable route to monoanions carrying three bulky organic groups attached to the amino N consisted in preparing amino derivs. from the appropriate primary or secondary amines and reacting these intermediate products with alkyl halides in alkaline aqueous PrOH solution The displacement of N2 by nitriles produced [1-RCNB10H9]- monoanions (R = CH3, Ph2CH) which proved to be thermally stable, but were easily hydrolyzed to [1-RCONH2B10H9]- monoanions.

ACCESSION NUMBER: 1999:310408 CAPLUS
DOCUMENT NUMBER: 131:38824
TITLE: Replacement of the nitrogen of [1-N2B10H9]- by amines or nitriles, a route to hydrophobic monoanions
AUTHOR(S): Naoufal, Daoud; Gruner, Bohumir; Bonnetot, Bernard; Mongeot, Henri
CORPORATE SOURCE: Laboratoire des Multimateriaux et Interfaces, UMR no 561, Laboratoire des Multimateriaux et Interfaces, UMR no 5615, Universite Claude Bernard Lyon I, Villeurbanne, F-69622, Fr.
SOURCE: Polyhedron (1999), 18(7), 931-939
CODEN: PLYHDE; ISSN: 0277-5387
PUBLISHER: Elsevier Science Ltd.
DOCUMENT TYPE: Journal
LANGUAGE: English
REFERENCE COUNT: 30 THERE ARE 30 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 93 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB The synthesis and characterization of a series of phenothiazines for possible use in photochemotherapy is reported. Oxidative amination of 10H-phenothiazine using anilines and iodine in THF led to a series of 3,7-bis(arylamino)-5-phenothiazinium salts. 4-Substituted primary anilines gave rise to a secondary amino functionality at positions 3- and 7- of the phenothiazine chromophores. The relative ease of deprotonation of these compds. to the corresponding quinone imines correlated well with the electronic properties of the 4-substituent in the original aniline. In vitro singlet oxygen yields for these derivs. were much lower than for the standard photosensitizer, methylene blue. The use of N-methylaniline did not lead to increased photosensitizing efficacy. However, the phenothiazines resulting from the use of benzylamines in place of anilines were more akin to new methylene blue N. All of the derivs. exhibited much greater lipophilicities than methylene blue.

ACCESSION NUMBER: 1999:296208 CAPLUS
DOCUMENT NUMBER: 131:60009
TITLE: Phenothiazine photosensitizers: part 2. 3,7-Bis(arylamino)phenothiazines
AUTHOR(S): Wainwright, Mark; Grice, Nicola J.; Pye, Lynnette E. C.
CORPORATE SOURCE: Photochemotherapy Group, Department of Applied Biology, University of Central Lancashire, Preston, PR1 2HE, UK
SOURCE: Dyes and Pigments (1999), 42(1), 45-51
CODEN: DYPIDX; ISSN: 0143-7208
PUBLISHER: Elsevier Science Ltd.
DOCUMENT TYPE: Journal
LANGUAGE: English
REFERENCE COUNT: 19 THERE ARE 19 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 94 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

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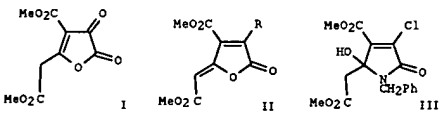

AB Reaction of (MeO2CCH2)2CO with (COCl)2 and MgCl2 as catalyst yielded 2,3-dioxo-2,3-dihydrofuran I, which is in equilibrium with tautomer II (R = OH; I/II = 1:2). Addition of SOCl2 to a mixture of I and II (R = OH) afforded 3-chloro-2(5H)-furanone II (R = Cl). The structure of II (R = Cl) was unequivocally established by x-ray diffraction. Ring opening of II (R = Cl) by nucleophilic attack with PhCH2NH2 at C(2) and subsequent recyclization led to racemic 3-chloro-5-hydroxy-2-oxo-2,5-dihydropyrrole III. According to single-crystal x-ray anal., III aggregates via stereospecific self-selection through H bonds to give chiroselectively the 1-dimensional strands ∞1[(S)-III] and ∞1[(R)-III].

ACCESSION NUMBER: 1999:161339 CAPLUS
DOCUMENT NUMBER: 130:267301
TITLE: Synthesis and aggregation of a 5-hydroxy-2,5-dihydropyrrole. Enantiomerically pure, one-dimensional strands via hydrogen bonds and chiroselective self organization
AUTHOR(S): Saalfrank, Rolf W.; Nachtrab, Jochen; Reck, Stephan; Hampel, Frank
CORPORATE SOURCE: Institut Organische Chemie, Universitaet Erlangen-Nuernberg, Erlangen, D-91054, Germany
SOURCE: Zeitschrift fuer Naturforschung, B: Chemical Sciences (1999), 54(2), 179-186
CODEN: ZNBSEN; ISSN: 0932-0776
PUBLISHER: Verlag der Zeitschrift fuer Naturforschung
DOCUMENT TYPE: Journal
LANGUAGE: German
OTHER SOURCE(S): CASREACT 130:267301
REFERENCE COUNT: 62 THERE ARE 62 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 95 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB Reaction between (Me2N)3P, CCl4, and RR1C:NOH [R = R1 = Me, Ph; R = Ph, 4-O2NC6H4, R1 = Me] gives RR1C:NOP+(NMe2)3 PF6-. These salts are solid and stable except if they are completely dehydrated. Their solns., in non-polar solvents like CHCl3, undergo Beckmann rearrangement at room temperature The kinetics and mechanism have been studied by NMR. The cationic intermediates formed in the rearrangement were trapped with amines to give amidines and a sugar hemiacetal to give a glycoside structure.

ACCESSION NUMBER: 1999:100571 CAPLUS
DOCUMENT NUMBER: 130:223035
TITLE: Beckmann rearrangement of OTDP salts of oximes of aromatic ketones and synthetic applications
AUTHOR(S): Thiebaut, Sylvie; Gerardin-Charbonnier, Christine; Selve, Claude
CORPORATE SOURCE: Laboratoire de Chimie Physique Organique et Colloidale, Universite Henri Poincare - Nancy I, NANCY VANDOEUVRE, 54506, Fr.
SOURCE: Tetrahedron (1999), 55(5), 1329-1340
CODEN: TETRAB; ISSN: 0040-4020
PUBLISHER: Elsevier Science Ltd.
DOCUMENT TYPE: Journal
LANGUAGE: French
REFERENCE COUNT: 22 THERE ARE 22 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 96 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB A compound, and method of making a compound, for use as a diagnostic or therapeutic pharmaceutical comprises at least one functionalized hydroxyalkyl phosphine donor group and one or more sulfur or nitrogen donor and a metal combined with the ligand. Preparation and characterization of ligands and e.g. 99mTc complexes are described. The compds. are useful for therapeutic and diagnostic radiopharmaceuticals.

ACCESSION NUMBER: 1999:42478 CAPLUS
DOCUMENT NUMBER: 130:92218
TITLE: Hydroxymethyl phosphine compounds, and preparation thereof, for use as diagnostic and therapeutic pharmaceuticals
INVENTOR(S): Katti, Kattesh V.; Karra, Srinivasa Rao; Berning, Douglas E.; Smith, C. Jeffrey; Volkert, Wynn A.; Ketring, Alan R.
PATENT ASSIGNEE(S): The Curators of the University of Missouri, USA
SOURCE: U.S., 34 pp., Cont.-in-part of U.S. Ser. No. 412,470, abandoned.
CODEN: USXXAM
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 3
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 5855867	A	19990105	US 1997-818080	19970314
CA 2215833	AA	19961003	CA 1996-2215833	19960307
US 5876693	A	19990302	US 1997-902829	19970730
US 6054115	A	20000425	US 1998-33928	19980303
CA 2277179	AA	19980924	CA 1998-2277179	19980305
WO 9841242	A1	19980924	WO 1998-US4318	19980305
W: AU, CA, JP				
RW: AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE				
AU 9865429	A1	19981012	AU 1998-65429	19980305
EP 1009447	A1	20000621	EP 1998-911487	19980305
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, FI				
JP 2001516360	T2	20010925	JP 1998-540558	19980305

PRIORITY APPLN. INFO.: US 1995-412470 B2 19950329
US 1997-818080 A3 19970314
US 1997-902829 A1 19970730
WO 1998-US4318 W 19980305
OTHER SOURCE(S): MARPAT 130:92218
REFERENCE COUNT: 36 THERE ARE 36 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 97 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
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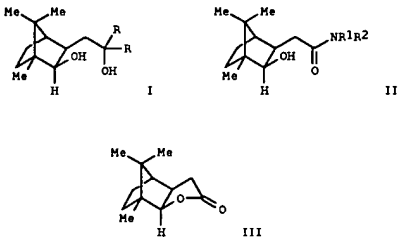

AB New enantiomerically pure 1,4-diols I (R = H, Ph) and 1,4-aminoalcs. II [R1 = Me, Et, Me2CHCH2, PhCH2; R2 = Et, Me2CHCH2, Ph, PhCH2, 1-naphthyl or R1R2 = (S)-2-(methoxymethyl)-1-pyrrolidinyl, morpholinyl] have efficiently been prepared in one and two steps, resp., from a com. available camphor derived exo fused lactone III. Using sterically hindered amines such as diisopropylamine, an aldol addition of two lactone mols. was observed and the stereochem. of the products was determined by X-ray crystallog.

ACCESSION NUMBER: 1999:24499 CAPLUS
DOCUMENT NUMBER: 130:168029
TITLE: New camphor derived chiral ligands for asymmetric synthesis
AUTHOR(S): Knollmuller, Max; Ferencic, Mathias; Gartner, Peter; Mereiter, Kurt; Noe, Christian R.
CORPORATE SOURCE: Institute of Organic Chemistry, Vienna University of Technology, Vienna, A-1060, Austria
SOURCE: Tetrahedron: Asymmetry (1998), 9(22), 4009-4020
CODEN: TASYE3; ISSN: 0957-4166
PUBLISHER: Elsevier Science Ltd.
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 130:168029
REFERENCE COUNT: 21 THERE ARE 21 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 98 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB HONR1R2 [R1, R2 = (un)substituted alkyl, (un)substituted aryl, (un)substituted aromatic hetero ring group], useful as developers for silver halide photog. materials and stabilizing agents for polymers (no data), are prepared by (a) preparation of mixts. containing HNR1R2 (R1, R2 = same as above), dehydrating agents., and organic solvents and (b) addition of Re catalysts and H2O2 to the mixts. Bis(2-methoxyethyl)amine was mixed with Mg(SO4)2 in AcOEt under ice-cooling, mixed with H2O2 and methyltrioxorhenium at 0-10° for 1.3 h to give a mixture containing 83% N,N-bis(2-methoxyethyl)hydroxylamine, which was treated with oxalic acid in acetone under ice-cooling for 30 min to give 74.0% N,N-bis(2-methoxyethyl)hydroxylamine oxalate.

ACCESSION NUMBER: 1998:795452 CAPLUS
DOCUMENT NUMBER: 130:81200
TITLE: Preparation of N,N-disubstituted hydroxylamines as developers for silver halide photographic materials and stabilizing agents for polymers
INVENTOR(S): Motoki, Masushi; Sato, Tadahisa
PATENT ASSIGNEE(S): Fuji Photo Film Co., Ltd., Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 11 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 10330342	A2	19981215	JP 1997-139517	19970529
US 6031130	A	20000229	US 1998-81943	19980521

PRIORITY APPLN. INFO.: JP 1997-139517 A 19970529
OTHER SOURCE(S): CASREACT 130:81200; MARPAT 130:81200

L12 ANSWER 99 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB Agents that can unwind duplexes and bind selectively to unfolded nucleic acids can be the basis of potential antiviral and anticancer drugs. These compds. could disrupt RNA secondary structures such as hairpin stem-loop conformations, which are important recognition sites for gene regulatory proteins that control viral replication. We describe here a new way to destabilize folded nucleic acid conformations by stabilizing unduplexed parts of the polymer, or single-stranded (ss) forms, which lead to destabilization effects of hitherto unknown magnitude with concns. as low as 50μM.

ACCESSION NUMBER: 1998:794794 CAPLUS
DOCUMENT NUMBER: 130:106569
TITLE: Supramolecular chemistry. Part 80. A new strategy for the destabilization of double-stranded nucleic acids by phenylalkylamine derivates
AUTHOR(S): Ali, Anmar; Gasiorek, Martin; Schneider, Hans-Jorg
CORPORATE SOURCE: FR 11.2 Organische Chemie, Universitat des Saarlandes, Saarbrucken, D-66041, Germany
SOURCE: Angewandte Chemie, International Edition (1998), 37(21), 3016-3019
CODEN: ACIEF5; ISSN: 1433-7851
PUBLISHER: Wiley-VCH Verlag GmbH
DOCUMENT TYPE: Journal
LANGUAGE: English
REFERENCE COUNT: 30 THERE ARE 30 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 100 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

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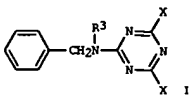

I

AB The triazine derivative I [X = OH, halo, VR1R2 (R1 = benzyl; R2 = benzyl, Ph)], and an electrophotog. toner therewith are claimed.

ACCESSION NUMBER: 1998:724195 CAPLUS
DOCUMENT NUMBER: 130:31150
TITLE: Dibenzylamino-substituted triazine derivative and electrophotographic toner therewith
INVENTOR(S): Aoyagi, Masayuki
PATENT ASSIGNEE(S): Nippon Kayaku Co., Ltd., Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 8 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 10298167	A2	19981110	JP 1997-122910	19970428
PRIORITY APPLN. INFO.:			JP 1997-122910	19970428

OTHER SOURCE(S): MARPAT 130:31150

L12 ANSWER 101 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB Reaction of pre-formed crystalline amides [(PhCH2)2NLi] and [Me2Al{N(CH2Ph)2] in the presence of pyridine results in the mixed metal complex [Me2Al{(PhCH2)2N}2Li·pyr] 1. Ab initio MO calcns. indicate formation of the bimetallic product is energetically favorable. The possible driving forces for the reaction are discussed using single crystal X-ray anal. for 1 and the pyridine solvate [{(PhCH2)2NLi·pyr}2] 7, in combination with theor. calcns. A major contributing factor in stabilization of the bimetallic compound was a reduction in steric crowding within the mixed metal base compared to the homometallic dialkylaluminium amide. In addition, complex 1 shows significant benzyl to lithium interactions which contribute to the overall bonding. Such interactions are unusual with donor solvent present as competing complexant.

ACCESSION NUMBER: 1998:723102 CAPLUS
DOCUMENT NUMBER: 130:209734
TITLE: Synthesis, characterization and a theoretical investigation of the formation of lithium dialkylaluminum amides
AUTHOR(S): Clegg, William; Liddle, Stephen T.; Henderson, Kenneth W.; Keenan, Fiona E.; Kennedy, Alan R.; McKeown, Arlene E.; Mulvey, Robert E.
CORPORATE SOURCE: Department of Pure and Applied Chemistry, University of Strathclyde, Glasgow, G1 1XL, UK
SOURCE: Journal of Organometallic Chemistry (1999), 572(2), 283-289
CODEN: JORCAI; ISSN: 0022-328X
PUBLISHER: Elsevier Science S.A.
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 130:209734
REFERENCE COUNT: 32 THERE ARE 32 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 102 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

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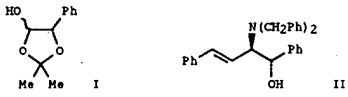

I II

AB Anti-β-amino alcs. RCH(NR1R2)CH(OH)R3 [R = (E)-PhC(Br):CH, (E)-PhCH:CH, 4-MeOC6H4, 2-thienyl, Bu, MeOCH2, 2-furyl, N-(tert-butoxycarbonyl)-2-pyrrolyl; R1 = Ph2CH, PhCH2; R2 = H, Me, PhCH2; R3 = HOCH2, HOCH(Me), HOCH(Ph), HOCH(CH2Bu)] are prepared in a single step with >99% de and in 39-88% yield from alkenyl or arylboronic acids RB(OH)2, amines R1R2NH, and α-hydroxyaldehydes R3CH(OH)CHO or 4-hydroxy-5-alkyl-1,3-dioxolanes. Enantiomerically pure α-hydroxyaldehydes such as (R)-glyceraldehyde provide anti-β-amino alcs. in >99% ee and >99% de. E.g., nonracemic dioxolane I, (E)-PhCH:CHB(OH)2, and HN(CH2Ph)2 react in EtOH at room temperature to give the enantiomeric pure amino alc. II in 88% yield. (R)-glyceraldehyde can be used as an α-hydroxyaldehyde to give access to novel amino acids by ruthenium oxidation of the amino diol product.

ACCESSION NUMBER: 1998:694160 CAPLUS
DOCUMENT NUMBER: 130:51998
TITLE: Highly Stereocontrolled One-Step Synthesis of anti-β-Amino Alcohols from Organoboronic Acids, Amines, and α-Hydroxy Aldehydes
AUTHOR(S): Petasis, Nicos A.; Zavialov, Ilia A.
CORPORATE SOURCE: Department of Chemistry Loker Hydrocarbon Research Institute, University of Southern California, Los Angeles, CA, 90089-1661, USA
SOURCE: Journal of the American Chemical Society (1998), 120(45), 11798-11799
CODEN: JACSAT; ISSN: 0002-7863
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 130:51998
REFERENCE COUNT: 68 THERE ARE 68 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 103 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
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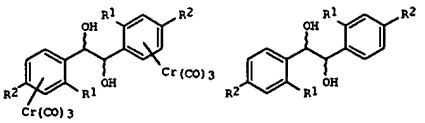


I

II

AB An intermol. pinacol coupling of the planar chiral tricarbonylchromium complexes of o-substituted benzaldehydes or benzaldimines with samarium(II) diiodide in THF produces exclusively threo 1,2-diols or 1,2-diamines in an optically pure form, while the corresponding racemic o-substituted benzaldehyde or benzaldimine chromium complexes give a mixture of threo and erythro pinacol coupling products in a various ratio depending upon the nature of o-substituent. Similarly, planar chiral 2-substituted ferrocenecarboxaldehydes and (dienal)Fe(CO)3 produce the corresponding 1,2-diols with high stereoselectivity. The generated transition metal-complexed ketyl radical intermediates are configurationally stable with restriction to a rotation about Cα-Cipso bond. Thus, pinacol coupling of benzaldehyde chromium complexes with SmI2 in THF gave chromium complexes I (R1 = H, Me, OMe, OPri, NMe2, Br, R2 = H; R1 = H, R2 = Me, OMe, Br), which on demetalation with I2 gave pinacols II.

ACCESSION NUMBER: 1998:652007 CAPLUS
DOCUMENT NUMBER: 130:25164
TITLE: Stereoselective pinacol coupling of planar chiral (benzaldehyde)Cr(CO)3, (benzaldimine)Cr(CO)3, ferrocenecarboxaldehyde and (dienal)Fe(CO)3 complexes with samarium diiodide
AUTHOR(S): Taniguchi, Nobukazu; Uemura, Motokazu
CORPORATE SOURCE: Dep. Chem., Fac. Integrated Arts Sci., Osaka Prefecture Univ., Sakai, Osaka, 599-8531, Japan
SOURCE: Tetrahedron (1998), 54(42), 12775-12788
CODEN: TETRAB; ISSN: 0040-4020
PUBLISHER: Elsevier Science Ltd.
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 130:25164
REFERENCE COUNT: 66 THERE ARE 66 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 104 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB A compound and method of making a compound for use as a diagnostic or therapeutic pharmaceutical comprises at least one functionalized hydroxyalkyl phosphine donor group and one or more S or N donor and a metal combined with the ligand. Thus, the preparation and coordination chemical of bis(hydroxymethyl)phosphines containing thioether donor groups, e.g., (HOCH2)2P(CH2)2S(CH2)3S(CH2)2P(CH2OH)2 and (HOCH2)2P(CH2)3S(CH2)3S(CH2)3P(CH2OH)2, and of N-donor analogs, with technetium or rhenium, are presented. The in vivo stability and biodistribution studies of the complexes in rat demonstrate the use of these complexes as radiopharmaceuticals, as diagnostic and therapeutic agents.

ACCESSION NUMBER: 1998:635679 CAPLUS
DOCUMENT NUMBER: 129:285209
TITLE: Hydroxymethyl phosphine compounds for use as diagnostic and therapeutic pharmaceuticals and method of making same
INVENTOR(S): Katti, Kattesh V.; Karra, Srinivasa Rao; Berning, Douglas E.; Smith, C. Jeffrey; Volkert, Wynn A.; Ketring, Alan R.
PATENT ASSIGNEE(S): The Curators of the University of Missouri, USA
SOURCE: PCT Int. Appl., 149 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 3
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 9841242	A1	19980924	WO 1998-US4318	19980305
W: AU, CA, JP				
RW: AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE				
US 5855867	A	19990105	US 1997-818080	19970314
CA 2277179	AA	19980924	CA 1998-2277179	19980305
AU 9865429	A1	19981012	AU 1998-65429	19980305
EP 1009447	A1	20000621	EP 1998-911487	19980305
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, FI				
JP 2001516360	T2	20010925	JP 1998-540558	19980305
PRIORITY APPLN. INFO.:			US 1997-818080 A	19970314
			US 1995-412470 B2	19950329
			WO 1998-US4318 W	19980305

OTHER SOURCE(S): MARPAT 129:285209
REFERENCE COUNT: 3 THERE ARE 3 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 105 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB The synthesis of five enzymically stable analogs of guanosine diphosphate (GDP) has been carried out. The pyrophosphate moiety was mimicked in turn by the malonate, the acetophosphonate, the phosphonoacetate, the methylene-bis-phosphonate, and the imidodiphosphate groups. All the compds. were prepared via the synthesis of a transient fully protected nucleoside diphosphate analog, and the final deprotection step was achieved by catalytic hydrogenolysis. The biol. properties of the compds. have been evaluated toward transducin, the G-protein of the visual photoreceptor. Three guanosine imidodiphosphate derivs. bearing a linker at different positions on the sugar and on the base were then prepared and evaluated, giving some insight into the GDP binding site of transducin.

ACCESSION NUMBER: 1998:619311 CAPLUS
DOCUMENT NUMBER: 129:316485
TITLE: Synthesis of Enzymically Stable Analogs of GDP for Binding Studies with Transducin, the G-Protein of the Visual Photoreceptor
AUTHOR(S): Vincent, Stephane; Grenier, Sonya; Valleix, Alain; Salesse, Christian; Lebeau, Luc; Mioskowski, Charles
CORPORATE SOURCE: Laboratoire de Synthese Bioorganique associe au CNRS Faculte de Pharmacie, Universite Louis Pasteur de Strasbourg, Illkirch, 67 401, Fr.
SOURCE: Journal of Organic Chemistry (1998), 63(21), 7244-7257
CODEN: JOCEAH; ISSN: 0022-3263
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
REFERENCE COUNT: 106 THERE ARE 106 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 106 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB This document discloses a process for producing an aryl carbamate of a high purity in a high yield by reacting a diaryl carbonate with an amine compound having one or more hydrogen atoms bonded to the N position in the presence of carboxylic acid(s) of the following general formulas R1CO2H and/or R2CO2H (wherein R1 represents an alkyl or cycloalkyl group having a carbon atom at the α-position, which is bonded to only one hydrogen atom, and R2 represents an alkyl group having a carbon atom at the α-position, which is not bonded to a hydrogen atom). Thus, a mixture of di-Ph carbonate 0.01 mol, aniline 0.012 mol, and pivalic acid 0.002 mol was heated at 75° for 4 h to give Ph N-phenylcarbamate (with 98.4% selectivity) in 93% yield.

ACCESSION NUMBER: 1998:568801 CAPLUS
DOCUMENT NUMBER: 129:189134
TITLE: Process for producing aryl carbamates
INVENTOR(S): Harada, Katsumasa; Sugise, Ryoji; Kashiwagi, Kohichi; Matsuura, Tsunao
PATENT ASSIGNEE(S): Ube Industries, Ltd., Japan
SOURCE: PCT Int. Appl., 75 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 9835936	A1	19980820	WO 1998-JP592	19980213
W: US				
RW: AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE				
JP 10316645	A2	19981202	JP 1997-129607	19970520
JP 10287638	A2	19981027	JP 1998-31628	19980213
JP 3480296	B2	20031215		
JP 10287639	A2	19981027	JP 1998-31629	19980213
JP 3508530	B2	20040322		
JP 10287640	A2	19981027	JP 1998-31630	19980213
EP 902014	A1	19990317	EP 1998-902760	19980213
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, FI				
US 6143917	A	20001107	US 1998-171076	19981013
PRIORITY APPLN. INFO.:			JP 1997-30459 A	19970214
			JP 1997-30460 A	19970214
			JP 1997-30461 A	19970214
			JP 1997-129607 A	19970520
			WO 1998-JP592 W	19980213

OTHER SOURCE(S): CASREACT 129:189134; MARPAT 129:189134
REFERENCE COUNT: 2 THERE ARE 2 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

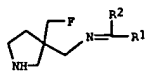
L12 ANSWER 107 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB A new pathway for n.c.a. 18F-labeling of biogenic arylalkylamines such as [18F]fluorononephedrine and [18F]fluorometaraminol (FMR) via nucleophilic aromatic substitution was developed. To overcome the problem of low specific activity, 18F-labeled arylalkylamines were synthesized by direct nucleophilic exchange with n.c.a. [18F]fluoride starting with a keto-activated aromatic system and consecutive chiral reduction of the keto-function. With regard to a stereoselective reduction of the CO group, several N-protected α-aminopropiophenones were prepared as model compds. to examine the influence of the protecting group on the radiochem. yield of a 18F-for-X substitution (X = F, Cl, NO2, N+Me3). Good radiochem. yields could be achieved using N-dibenzyl- or acetyl-protected compds. The para-position of the leaving group provided higher radiochem. yields than the ortho-position in the case of the 18F-for-19F substitution. The less basic oxalate/cryptate system does not increase the radiochem. yields. 18F-fluorination of the nitro compound failed because the precursor was not stable under labeling conditions. The best results of n.c.a. 18F-fluorination were obtained using the N+Me3 leaving group in para-position (.apprx.50% radiochem. yield), however, a selective quaternization of the dimethylaniline group was only possible when using the N,N-dibenzylated derivative The n.c.a. labeling of 4-[18F]fluorononephedrine and 4-[18F]fluorometaraminol was finally performed via 18F-for-NMe3 substitution on 4-(2-N,N-dibenzylaminopropionyl)phenyl-1-N,N,N-trimethylammonium triflate and 4-(2-N,N-dibenzylaminopropionyl)-2-benzyloxyphenyl-1-N,N,N-trimethylammonium triflate, resp. The precursor of 4-[18F]fluorometaruninol was synthesized in an 11-step reaction sequence and characterized with IR and 1H-NMR. The formation of the threo-isomer of n.c.a. 4-[18F]fluorononephedrine was achieved by reduction of n.c.a. 2-N,N-dibenzylamino-1-(4-[18F]fluorophenyl)propan-1-one using NaBH4 in MeOH. The radiochem. yield was .apprx.20% after debenzylation using HCO2NH4 and Pd/C. The formation of erythro-4-[18F]fluorononephedrine and -4-[18F]fluorometaraminol was accomplished with BH3.THF in the presence of 2-N,N-dibenzylamino-1-(4-[18F]fluoro-phenyl)propan-1-one and 2-N,N-dibenzylamino-1-(4-[18F]fluoro-3-benzyloxyphenyl)propan-1-one, resp. The ratio of erythro- to threo-isomer was 4:1. The radiochem. yield of erythro-4-[18F]fluorononephedrine and erythro-4-[18F]fluorometaraminol after deprotection was 15-20% with a specific activity of .apprx.74 GBq/μmol (2 Ci/μmol).

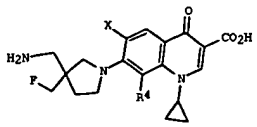
ACCESSION NUMBER: 1998:494230 CAPLUS
DOCUMENT NUMBER: 129:161384
TITLE: No-carrier-added 18F-labeling of arylalkylamines with norephedrine and metaraminol as examples
AUTHOR(S): Ermert, Johannes
CORPORATE SOURCE: Inst. Nuklearchemie, Forschungszentrum Juelich G.m.b.H., Juelich, D-52425, Germany
SOURCE: Berichte des Forschungszentrums Juelich (1998), Juel-3499, 1-136
CODEN: FJBEE5; ISSN: 0366-0885
DOCUMENT TYPE: Report
LANGUAGE: German

L12 ANSWER 108 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

GI



I



II

AB Characterized is a process for preparation of the title compds. (I; R1, R2 = H, alkyl, aryl, etc.) as intermediates for the synthesis of quinolone-carboxylic acid derivs. (II; X = halo, R4 = OMe, halo) which are useful as antibacterial agents. The process comprises reacting an aminomethyl group on a pyrrolidine ring with an aldehyde or a ketone to temporarily protect the aminomethyl group in the form of a Schiff's base, conducting a condensation reaction with a skeleton, and removing the protective group. According to this process, intended compds. can be produced in a high purity and a high yield in a simple manner without producing any byproduct. Thus, (S)-(+)-3-aminomethyl-3-fluoromethylpyrrolidine (preparation given) was reacted with C6H5CHO to give 100% I (R1 = Ph, R2 = H), which was further reacted with quinolone-carboxylic acid derivative to give II (X = F, R4 = OMe).

ACCESSION NUMBER: 1998:197499 CAPLUS
DOCUMENT NUMBER: 128:204909
TITLE: Process for producing pyrrolidine derivatives as intermediates for the synthesis of quinolone-carboxylic acid derivatives
INVENTOR(S): Okuda, Hirofumi; Ikebe, Tsuguo; Ohe, Takanori; Tsuruda, Mineo
PATENT ASSIGNEE(S): Yoshitomi Pharmaceutical Industries, Ltd., Japan
SOURCE: PCT Int. Appl., 43 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 9812191	A1	19980326	WO 1996-JP2664	19960917

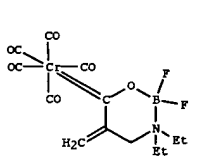
L12 ANSWER 108 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)

W: AL, AU, BA, BB, BG, BR, CA, CN, CU, CZ, EE, GE, HU, IL, IS, KR, LC, LK, LR, LT, LV, MG, MK, MN, MX, NO, NZ, PL, RO, SG, SI, SK, TR, TT, UA, US, UZ, VN, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM
RW: KE, LS, MW, SD, SZ, UG, AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG

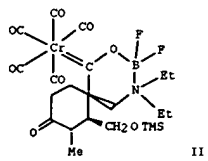
AU 9669462 A1 19980414 AU 1996-69462 19960917
PRIORITY APPLN. INFO.: WO 1996-JP2664 W 19960917
OTHER SOURCE(S): CASREACT 128:204909; MARPAT 128:204909
REFERENCE COUNT: 3 THERE ARE 3 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 109 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

GI



I



II

AB A new type of cyclic amino-functionalized s-cis boroxyvinylcarbene complex of Group 6 metals was synthesized, e.g. I. These complexes underwent Diels-Alder-type reactions with 2-amino 1,3-dienes that proceeded with complete regioselectivity and high exo or endo diastereoselectivity, which is highly dependent on the nature of the substituents on the diene. When chiral 2-amino-5-alkoxy dienes derived from (S)-prolinol benzyl or Me ether were used, an exclusive exo and highly diastereofacially selective [4 + 2] cycloaddn. was achieved, affording spiro carbene complexes with three contiguous stereogenic centers and a high level of enantiomeric purity, e.g. II. Removal of the Cr(CO)5 fragment and the BF2 group provided an entry to α,α-branched β-amino aldehydes or β-amino acids. The stable form of an amino-substituted hydroxycarbene complex of Cr was characterized by x-ray diffraction.

ACCESSION NUMBER: 1998:150278 CAPLUS
DOCUMENT NUMBER: 128:217476
TITLE: Cyclic BF2 Adducts of Functionalized Fischer Vinylcarbene Complexes: Preparation and Stereoselective Diels-Alder Reactions with 2-Amino 1,3-Dienes
AUTHOR(S): Barluenga, Jose; Canteli, Rosa-Maria; Florez, Josefa; Garcia-Granda, Santiago; Gutierrez-Rodriguez, Angel; Martin, Eduardo
CORPORATE SOURCE: Instituto Universitario de Quimica Organometalica Enrique Moles Unidad Asociada al CSIC and Departamento de Quimica Fisica y Analitica, Universidad de Oviedo, Oviedo, 33071, Spain
SOURCE: Journal of the American Chemical Society (1998), 120(11), 2514-2522
CODEN: JACSAT; ISSN: 0002-7863
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 128:217476
REFERENCE COUNT: 126 THERE ARE 126 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 110 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB A two-step method for preparing tert-butylsulfonamides from primary and secondary amines is described. E.g., treating (PhCH2)2NH with Me3CSOCl gave sulfinamide (PhCH2)2NSOCMe3, which was oxidized by either m-CPBA or RuCl3/NaIO4 to give (PhCH2)2NSO2CMe3. The Bus derivs. are stable to strong bases and metalation conditions and are cleaved to the parent amines by mild acidic solvolysis. Secondary sulfonamides can be selectively cleaved in the presence of primary ones.

ACCESSION NUMBER: 1997:724086 CAPLUS
DOCUMENT NUMBER: 128:22499
TITLE: tert-Butylsulfonyl (Bus), a New Protecting Group for Amines
AUTHOR(S): Sun, Pu; Weinreb, Steven M.; Shang, Maoyu
CORPORATE SOURCE: Department of Chemistry, Pennsylvania State University, University Park, PA, 16802, USA
SOURCE: Journal of Organic Chemistry (1997), 62(24), 8604-8608
CODEN: JOCEAH; ISSN: 0022-3263
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 128:22499
REFERENCE COUNT: 12 THERE ARE 12 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 111 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB Primary amines can be converted in high yield into N,N-dibenzyl formamidines under mild conditions. The N,N-dibenzyl formamidine group was found to be effective as a protective group for primary amines as it is stable to a variety of conditions and can be removed by catalytic hydrogenation.

ACCESSION NUMBER: 1997:706262 CAPLUS
DOCUMENT NUMBER: 128:13386
TITLE: N,N-Dibenzyl formamidine as a new protective group for primary amines
AUTHOR(S): Vincent, Stephane; Mons, Stephane; Lebeau, Luc; Mioskowski, Charles
CORPORATE SOURCE: Laboratoire de Synthese Bioorganique associe au CNRS - Faculte de Pharmacie, Universite Louis Pasteur de Strasbourg, Illkirch, 67 401, Fr.
SOURCE: Tetrahedron Letters (1997), 38(43), 7527-7530
CODEN: TELEAY; ISSN: 0040-4039
PUBLISHER: Elsevier
DOCUMENT TYPE: Journal
LANGUAGE: English
REFERENCE COUNT: 15 THERE ARE 15 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 112 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB Dihydrogen reduction of nitroalkanes, aliphatic and aromatic nitriles and ketones to their corresponding saturated products was successively achieved in DMF medium using polystyrene based acetato-bridged orthometalated Schiff base complexes of palladium(II) as catalysts, at 80-130°C and 6.0-14.0×103 (kN m-2) of PH2. The acetato-bridged Schiff base complexes are the catalyst precursors and the actual catalysts are the corresponding hydrogen activated orthometalated complexes with the acetate bridge replaced by H and DMF. The immobilization of the palladium(II) complexes in the polymer matrix slightly decreased their catalytic activities on the basis of metal content but improved the chemical and thermal stabilities and product selectivities relative to those of the corresponding homogeneous ones. The same specimen of the catalyst can be used repeatedly for the reduction of different substrates and stored for a long time without suffering any appreciable loss of activity. XPS data suggest the presence of palladium(II) in the fresh and used catalyst and kinetic studies indicate 1st order rate dependence on palladium(II) content, second order on PH2, and independent of substrate concentration A plausible mechanistic route has been suggested on the basis of kinetic data and exptl. observations.

ACCESSION NUMBER: 1997:541734 CAPLUS
DOCUMENT NUMBER: 127:262302
TITLE: Use of polystyrene bound orthometalated Schiff base complexes of palladium(II) as catalysts for the dihydrogen reduction of nitroalkanes, nitriles and ketones
AUTHOR(S): Islam, S. M.; Palit, B. K.; Mukherjee, D. K.; Saha, C. R.
CORPORATE SOURCE: Department of Chemistry, Indian Institute of Technology, Kharagpur 721302 W.B., India
SOURCE: Journal of Molecular Catalysis A: Chemical (1997), 124(1), 5-20
CODEN: JMCCF2; ISSN: 1381-1169
PUBLISHER: Elsevier
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 127:262302
REFERENCE COUNT: 43 THERE ARE 43 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 113 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB Coal-water slurry compns. with improved long-term storage stability and fluidity contain (A) water-soluble polymers, e.g., aliphatic diene-series (co)polymer sulfonates, (B) aromatic amine compds. selected from ≥1 of diphenylamine, benzylamine, and dibenzylamine, (C) coal, and (D) water as major component.

ACCESSION NUMBER: 1997:502085 CAPLUS
DOCUMENT NUMBER: 127:111109
TITLE: Coal-water slurry compositions
INVENTOR(S): Betsusho, Keiichi; Nagatsuka, Tomio; Ishikawa, Katsuhiro; Takano, Shinji; Manome, Kazuo
PATENT ASSIGNEE(S): Japan Synthetic Rubber Co., Ltd., Japan; Japan Communication Co., Ltd.
SOURCE: Jpn. Kokai Tokkyo Koho, 7 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 09143483	A2	19970603	JP 1995-323567	19951120
PRIORITY APPLN. INFO.:			JP 1995-323567	19951120

L12 ANSWER 114 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB Stabilized carbon and nitrogen nucleophiles can be efficiently allylated in a regioselective manner using allylic sulfoximines and palladium(0) catalysis.
ACCESSION NUMBER: 1997:349355 CAPLUS
DOCUMENT NUMBER: 127:65550
TITLE: Palladium(0) catalyzed allylation reactions with racemic and enantiomerically pure allylic sulfoximines
AUTHOR(S): Pyne, Stephen G.; O'meara, Gareth; David, Dorothy M.
CORPORATE SOURCE: Department of Chemistry, University of Wollongong, Wollongong, 2522, Australia
SOURCE: Tetrahedron Letters (1997), 38(20), 3623-3626
CODEN: TELEAY; ISSN: 0040-4039
PUBLISHER: Elsevier
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 127:65550
REFERENCE COUNT: 21 THERE ARE 21 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 115 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB A new methodol. for solution-phase chemical library synthesis and purifn. is described. This approach applies fundamental properties of complementary mol. reactivity and recognition (CMR/R) as the basis for a general purification strategy. Specifically, parallel solution-phase reactions are purified by resins containing mol. recognition or mol. reactivity functionalities complementary to those of solution-phase reactants, reagents, and byproducts. When used in sequential or simultaneous combinations, various CMR/R resins remove excess reactants, reagents, and byproducts from solution-phase reaction products, which are isolated in purified form by filtration. Where reactions involve the need to remove byproducts or reagents that do not inherently contain sequestrable functionality, sequestration can be effected by the design and use of tagged reactants or reagents containing artificially imparted mol. recognition functionality. An extension of this methodol. utilizes CMR/R resins as the "quench phase" instead of a liquid-phase workup commonly used in other library purification strategies. Hence, the essential features of complementary mol. reactivity or mol. recognition required for reaction workup are expressed on resins. The CMR/R library purification strategy is general and highly amenable to automation. Examples are illustrated with amine acylations, the Moffatt oxidation, and the reaction of organometallics with carbonyl compds.
ACCESSION NUMBER: 1997:324829 CAPLUS
DOCUMENT NUMBER: 126:343148
TITLE: Chemical Library Purification Strategies Based on Principles of Complementary Molecular Reactivity and Molecular Recognition
AUTHOR(S): Flynn, Daniel L.; Crich, Joyce Z.; Devraj, Rajesh V.; Hockerman, Susan L.; Parlow, John J.; South, Michael S.; Woodard, Scott
CORPORATE SOURCE: Section of Parallel Medicinal and Combinatorial Chemistry, Searle Discovery Research, St. Louis, MO, 63167, USA
SOURCE: Journal of the American Chemical Society (1997), 119(21), 4874-4881
CODEN: JACSAT; ISSN: 0002-7863
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
REFERENCE COUNT: 29 THERE ARE 29 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 116 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB The title compds. were synthesized and tested for antibacterial activities in comparison with typical fluoroquinolones. An (S)-3-aminomethyl-3-fluoromethyl derivative (Y-688) was confirmed to be optimal because of being most active especially against Gram-pos. bacteria, including fluoroquinolone-resistant strains. Y-688 showed high photostability.
ACCESSION NUMBER: 1997:188925 CAPLUS
DOCUMENT NUMBER: 126:251060
TITLE: Synthesis and structural optimization of 7-(3,3-disubstituted-1-pyrrolidinyl)-1-cyclopropyl-6-fluoro-1,4-dihydro-8-methoxy-4-oxo-3-quinolinecarboxylic acids as antibacterial agents
AUTHOR(S): Kitani, Hiroyuki; Kuroda, Tsuyoshi; Moriguchi, Akihiko; Ao, Hideki; Hirayama, Fumihiro; Ikeda, Yoshifumi; Kawakita, Takeshi
CORPORATE SOURCE: Research Laboratories, Yoshitomi Pharmaceutical Industries, Ltd., Fukuoka, 871, Japan
SOURCE: Bioorganic & Medicinal Chemistry Letters (1997), 7(5), 515-520
CODEN: BMCLE8; ISSN: 0960-894X
PUBLISHER: Elsevier
DOCUMENT TYPE: Journal
LANGUAGE: English
REFERENCE COUNT: 7 THERE ARE 7 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 117 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB The oxidation of organic substrates catalyzed by 'sandwich' type transition metal substituted polyoxometalates of the general formula, NaxM2Zn3W19O68, (M = Ru, Mn, Zn, Pd, Pt, Co, Fe, Rh) was examined in three different reaction media. The manganese analog was dissolved in a 1,2-dichloroethane phase using a lipophilic quaternary ammonium counter cation. Various organic substrates were oxidized with 30% aqueous H2O2. Alkenes reactivity increased as a function of the nucleophilicity of the double bond, but decreased as a function of steric crowding in the cyclohexene series. Alkenols with primary hydroxyl groups reacted chemo- and stereoselectively to form the corresponding epoxy alcs. On the other hand, alkenols with secondary hydroxyl units did not react chemoselectively; both ketones and epoxy alcs. were formed. Diols were oxidized in most cases to ketols, except for 1,4-butanediol which yielded γ-butyrolactone. Secondary amines yielded hydroxyl amines except for piperidine which reacted with the solvent. A manganese containing catalyst supported on a functionalized silica particle was as active and selective as the organic solvent containing biphasic system for the oxidation of alkenes and alkenols. Reactions were also carried out by dissolving NaxM2Zn3W19O68 in aqueous solns. of 30% H2O2, 70% t-butylhydroperoxide or 0.02 M potassium persulfate in the absence of solvent. Hydrogen peroxide degraded all the TMSP compds. One degradation product was an effective and chemo- and stereoselective catalyst for the epoxidn. of primary alkenols. In alc. oxidation only the ruthenium precursor was active. For oxidns. with 70% t-butylhydroperoxide all compds. were stable but only the Na11Ru2Zn3W19O68 compound was active. Alcs. were oxidized selectively, however, alkenols yielded a mixture of products. With persulfate, some catalytic effects were observed in double bond oxidation
ACCESSION NUMBER: 1997:138267 CAPLUS
DOCUMENT NUMBER: 126:268857
TITLE: Catalytic oxidation with hydrogen peroxide catalyzed by 'sandwich' type transition metal substituted polyoxometalates
AUTHOR(S): Neumann, Ronny; Khenkin, Alexander M.; Juwiler, David; Miller, Hagit; Gara, Mohammad
CORPORATE SOURCE: Casali Institute of Applied Chemistry, Graduate School of Applied Science, The Hebrew University of Jerusalem, Jerusalem, 91904, Israel
SOURCE: Journal of Molecular Catalysis A: Chemical (1997), 117(1-3, Proceedings of the 6th International Symposium on the Activation of Dioxygen and Homogeneous Catalytic Oxidation, 1996), 169-183
CODEN: JMCCF2; ISSN: 1381-1169
PUBLISHER: Elsevier
DOCUMENT TYPE: Journal
LANGUAGE: English
REFERENCE COUNT: 39 THERE ARE 39 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 118 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB Enantiomerically pure 1,2-diamines are prepared by intermol. pinacol coupling of planar chiral (benzaldimine)Cr(CO)3 complexes with SmI2.
ACCESSION NUMBER: 1997:110803 CAPLUS
DOCUMENT NUMBER: 126:250948
TITLE: Synthesis of enantiomerically pure 1,2-diamines by reductive coupling of tricarbonyl(benzaldimine)chromium complexes
AUTHOR(S): Taniguchi, Nobukazu; Uemura, Motokazu
CORPORATE SOURCE: Fac. Integrated Arts Sciences, Osaka Prefecture Univ., Sakai, 593, Japan
SOURCE: Synlett (1997), (1), 51-53
CODEN: SYNLES; ISSN: 0936-5214
PUBLISHER: Thieme
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 126:250948

L12 ANSWER 119 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB The condensation of α-unsatd. aldehydes with benzotriazole and secondary amines affords α-benzotriazolylalkenylamines that exist in solution as mixts. of the corresponding benzotriazol-1-yl and benzotriazol-2-yl isomers resulting from their rapid dissociation into iminium cations and the benzotriazolyl anion. The reduction of these adducts with samarium diiodide (SmI2) takes place with formation of the benzotriazolyl anion and α-amino alkenyl radicals that undergo 5- or 6-exo-trig cyclizations leading to substituted cycloalkyl- or cycloheteroalkylamines. The presence of an electron-withdrawing substituent in the alkene subunit is required for efficient cyclizations. The formation of cyclopentylamines takes place with unusually high 1,5-cis selectivity (hex-5-enyl radical numbering), and the presence of a 2- or 4-Me substituent also imparts high 1,2- or 1,4-trans stereoinduction, resp. The corresponding six-membered rings, however, are formed with low diastereoselectivity. Semiempirical calcns. performed on model systems suggest that a stabilizing secondary orbital interaction between the amino group and the electron-deficient alkene might in part account for the enhanced cis-selectivity encountered.
ACCESSION NUMBER: 1997:88592 CAPLUS
DOCUMENT NUMBER: 126:143908
TITLE: Diastereoselective Synthesis of Cycloalkylamines by Samarium Diiodide-Promoted Cyclizations of α-Amino Radicals Derived from α-Benzotriazolylalkenylamines
AUTHOR(S): Aurrecoechea, Jose M.; Lopez, Beatriz; Fernandez, Alvaro; Arrieta, Ana; Cossio, Fernando P.
CORPORATE SOURCE: Facultad de Ciencias, Universidad del Pais Vasco, Bilbao, 48080, Spain
SOURCE: Journal of Organic Chemistry (1997), 62(4), 1125-1135
CODEN: JOCEAH; ISSN: 0022-3263
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 126:143908
REFERENCE COUNT: 107 THERE ARE 107 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 120 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB Phenylglycine (Phg) can be protected by treatment with an aqueous suspension of benzothiazole-2-sulfonyl chloride (Bts-Cl, betsyl chloride) or 5-methyl-1,3,4-thiadiazole-2-sulfonyl chloride (Ths-Cl, thisyl chloride) at pH 9.5-10.5 (NaOH-H2O) to give Bts-Phg-OH and Ths-Phg-OH. Reaction with thionyl chloride affords the corresponding N-protected acid chlorides and rapid coupling with representative amino acid esters is possible under two phase aqueous conditions. Minimal Phg racemization occurs in the coupling step with the hindered HZNCMe2CO2Me (H-Aib-OMe) substrate (99.8% product ee). The betsyl or thisyl groups can be removed reductively without measurable change (<0.15 de) in diastereomeric purity in the Phg-containing dipeptides using 50% H3PO2 in THF/H2O at 50-65° or in DMF at room temperature, and also with Zn/HOAc-EtOH. Other reducing agents such as Na2S2O4 or NaHSO3 could also be used for deprotection, but some epimerization of the Phg residue was detected. The 50% H3PO2/DMF cleavage method was used to deprotect Bts-Trp-Met-Asp-Phe-NH2 to the cholecystokinin C-terminal tetrapeptide at rt.
ACCESSION NUMBER: 1996:619209 CAPLUS
DOCUMENT NUMBER: 126:19202
TITLE: Heteroarene-2-sulfonyl chlorides (BtsCl; ThsCl): reagents for nitrogen protection and >99% racemization-free phenylglycine activation with SOCl2
AUTHOR(S): Vedejs, Edwin; Lin, Shouzhong; Klapars, Artis; Wang, Jiabing
CORPORATE SOURCE: Chemistry Department, University of Wisconsin, Madison, WI, 53706, USA
SOURCE: Journal of the American Chemical Society (1996), 118(40), 9796-9797
CODEN: JACSAT; ISSN: 0002-7863
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 126:19202
REFERENCE COUNT: 38 THERE ARE 38 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L12 ANSWER 121 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB A very simple self-assembling system, which produces inclusion complexes with pseudorotaxane geometries, is described. The self-assembly of eight pseudorotaxanes with a range of stoichiometries - 1:1, 1:2, 2:1, and 2:2 (host:guest) - has been achieved. These pseudorotaxanes self-assemble from readily available components - well-known crown ethers, such as dibenzo[24]crown-8 and bis-p-phenylene[34]crown-10, and secondary dialkylammonium hexafluorophosphate salts, such as (PhCH2)2-NH2+PH6- and (Bu)2NH2+PF6- - and have been characterized not only in the solid state, but also in solution and in the "gas phase". The pseudorotaxanes are stabilized largely by hydrogen-bonding interactions and, in some instances, by aryl-aryl interactions.
ACCESSION NUMBER: 1996:377639 CAPLUS
DOCUMENT NUMBER: 125:167944
TITLE: Molecular meccano. 6. Pseudorotaxanes formed between secondary dialkylammonium salts and crown ethers
AUTHOR(S): Ashton, Peter R.; Chrystal, Ewan J. T.; Glink, Peter T.; Menzer, Stephan; Schiavo, Cesare; Spencer, Neil; Stoddart, J. Fraser; Tasker, Peter A.; White, Andrew J. P.; Williams, David J.
CORPORATE SOURCE: Sch. Chem., Univ. Birmingham, Edgbaston, Birmingham, B15 2TT, UK
SOURCE: Chemistry--A European Journal (1996), 2(6), 709-728
Published in: Angew. Chem., Int. Ed. Engl., 35(11)
CODEN: CEUJED; ISSN: 0947-6539
PUBLISHER: VCH
DOCUMENT TYPE: Journal
LANGUAGE: English

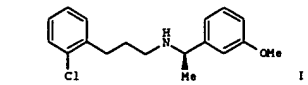
L12 ANSWER 122 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB In an attempt to develop a method for the general preparation of 1-alkenesulfenamides, some N,N-bis(trimethylsilyl)-1-alkenesulfenamides, e.g. (E)-BuCH:CHSN(SiMe3)2, were converted to a number of nitrogen functionalized analogs through desilylation and acylation procedures. Mono- and dibenzoylated derivs. (E)-BuCH:CHSNHCOPh and (E)-BuCH:CHSN(COPh)2 did not undergo transamination reactions with simple amines. Transamination reactions could be achieved once N,N-bis(trimethylsilyl)-1-alkenesulfenamides were converted to thiophthalimides, e.g. (E)-BuCH:CHSR (R = phthalimido). The transamination products, e.g. (E)-BuCH:CHSNHCH2Ph, are unstable to chromatog., but could be oxidized to 1-alkenesulfonamides using MCPBA. Some of the sulfenamides may be stable to distillation 3-(Alkenylthioimino)phthalides, isomers of thiophthalimides, also react with amines, but the process of ring opening accompanies transamination.

ACCESSION NUMBER: 1996:342089 CAPLUS
DOCUMENT NUMBER: 125:57526
TITLE: Transamination Studies on N-(1-Alkenylthio)phthalimides and Related Compounds. Synthesis of 1-Alkenesulfenamides and 1-Alkenesulfonamides
AUTHOR(S): Refvik, Mitchell D.; Schwan, Adrian L.
CORPORATE SOURCE: Guelph-Waterloo Centre for Graduate Work in Chemistry, University of Guelph, Guelph, ON, N1G 2W1, Can.
SOURCE: Journal of Organic Chemistry (1996), 61(13), 4232-4239
CODEN: JOCEAH; ISSN: 0022-3263
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 125:57526

L12 ANSWER 123 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

GI



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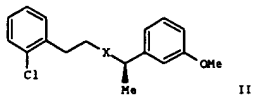

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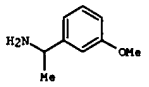
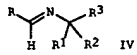


III



IV

AB A method of making the calcimimetic drug (R)-N-[1-(3-methoxyphenyl)ethyl]-3-(2-chlorobenzene)propanamine (I) involves reduction of amide or imine precursors II [X = CONH or CH:N] with an appropriate reducing agent. II is made from (R)-3-methoxy-α-methylbenzylamine [(R)-III]. Also disclosed is a method of condensing a nitrile with a primary or secondary amine to form an imine. This method involves reduction of a nitrile with DIBAL, and then reaction of the resultant compound with a primary or secondary amine to form the imine. The process is especially useful for producing enantiomerically pure chiral imines, and, ultimately, amines. Typical imines have formula IV [R, R1, R2, R3 independently = H, (un)substituted alkyl, aryl, aralkyl]. For example, (±)-III was prepared, then resolved using (R)-(-)-mandelic acid to give enantiomerically pure (R)-III in 83% yield. Then, 2-ClC6H4CH2CN was reduced with DIBAL in CH2Cl2, and treated with (R)-III at -78°, to give II [X = CH:N], which was reduced in situ with NaBH4 and EtOH, to give I in 76% yield.

ACCESSION NUMBER: 1996:332387 CAPLUS
DOCUMENT NUMBER: 125:10354
TITLE: Method of making a benzylpropanamine
INVENTOR(S): Vanwagenen, Bradford C.; Duff, Steven R.; Nelson, William A.; D'Ambra, Thomas E.
PATENT ASSIGNEE(S): NPS Pharmaceuticals, Inc., USA
SOURCE: PCT Int. Appl., 30 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 9602492	A1	19960201	WO 1995-US9081	19950714
W: AM, AT, AU, BB, BG, BR, BY, CA, CH, CN, CZ, DE, DK, EE, ES, FI, GB, GE, HU, IS, JP, KE, KG, KP, KR, KZ, LK, LR, LT, LU, LV, MD, MG, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, TJ, TM, TT				
RW: KE, MW, SD, SZ, UG, AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG				
US 5504253	A	19960402	US 1994-276214	19940715
US 5648540	A	19970715	US 1995-446491	19950522
AU 9531017	A1	19960216	AU 1995-31017	19950714
US 5633404	A	19970527	US 1996-639935	19960419

PRIORITY APPLN. INFO.: US 1994-276214 A 19940715
US 1995-446491 A3 19950522
WO 1995-US9081 W 19950714
OTHER SOURCE(S): CASREACT 125:10354; MARPAT 125:10354

L12 ANSWER 124 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB The content of wastewater resulting from the manufacture of rubber antioxidants and accelerators by a factory situated in the Ebro basin (Spain) has been determined using gas chromatog.-mass spectrometry (GC-MS) and gas chromatog.-flame ionization detection (GC-FID). The change in the pollutants was studied in the riverbed via two modules which continuously gathered pollutants on various solid supports (activated carbon and XAD-2 resins). These modules were located in Bocal Station, lying 100 km downstream from the factory and in the Zaragoza water supply. Forty-six different compds. were identified at Bocal Station, the majority resulting from the production of rubber additives. Due to the biol. stability of different waste substances and to the toxic nature of some, we studied their reactions when subjected to chemical oxidation using ozone.

ACCESSION NUMBER: 1996:313368 CAPLUS
DOCUMENT NUMBER: 125:17952
TITLE: Wastewater from the manufacture of rubber vulcanization accelerators: characterization, downstream monitoring and chemical treatment
AUTHOR(S): Puig, A.; Ormad, P.; Roche, P.; Sarasa, J.; Gimeno, E.; Ovelleiro, J. L.
CORPORATE SOURCE: Confederacion Hidrografica del Ebro, Po. de Sagasta 24-28, Zaragoza, 50006, Spain
SOURCE: Journal of Chromatography, A (1996), 733(1 + 2), 511-522
CODEN: JCRAEY; ISSN: 0021-9673
PUBLISHER: Elsevier
DOCUMENT TYPE: Journal
LANGUAGE: English

L12 ANSWER 125 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
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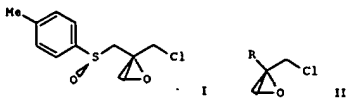

AB The reactivities of (RS)-1-chloro-3-p-tolylsulfinyl acetone towards diazomethane and of the resulting diastereoisomeric 1-chloromethyl-1-sulfinylmethyl oxirane I towards O-, N- and C-centered nucleophiles are investigated. The synthesis of differently functionalized homochiral chlorinated sulfur-free oxiranes (R)-II (R = CHO), (S)-II (R = CH2OH) and (R)-II (R = CO2H) has been accomplished in good chemical yields.

ACCESSION NUMBER: 1996:144591 CAPLUS
DOCUMENT NUMBER: 124:316885
TITLE: Synthesis and reactions of enantiomerically pure chloromethyl oxiranes
AUTHOR(S): Abrate, Francesca; Bravo, Pierfrancesco; Frigerio, Massimo; Viani, Fiorenza; Zanda, Matteo
CORPORATE SOURCE: Dip. Chim., Politec. Milano, Milan, I-20131, Italy
SOURCE: Tetrahedron: Asymmetry (1996), 7(2), 581-94
CODEN: TASYE3; ISSN: 0957-4166
PUBLISHER: Elsevier
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 124:316885

L12 ANSWER 126 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
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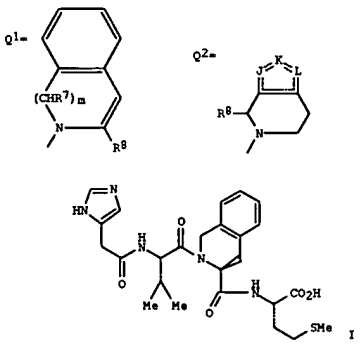

AB The title compds. G1-NR1-CA1R2-G [I; G = G2CONR3CA2R4G3, NR3(CH2)qQ, Q1, Q2; G1 = G4(CH2)nY, G4(CH2)nCH[(CH2)pNR5R6]Y, Q1, Q2, NR10CHQ3; wherein J, K, L = N, NR9, O, S, CR10, with the provisos that only one of the groups J, K and L can be O or S, and at least one of the groups J or L must be N, NR9, O or S to form a fused 5-membered heterocyclic ring; the bond between J and K or K and L may also form one side of a Ph ring fused to the 5-membered heterocyclic ring; Q = aryl; Q3, A1, A2 = H, (un)substituted alkyl or Ph; G3 = R11, CO2R11, CONR11R12, 5-tetrazolyl, CON(R13)OR11, CONHSO2R14, CH2OR11; G4 = 1-, 2-, 4- or 5-imidazolyl optionally substituted, at any of the available position or positions on the ring, with halo, C1-20 (un)substituted alkyl , alkoxy, aryl, aralkyl, OH, alkanoyl, alkanoyloxy, NH2, alkylamino, dialkylamino, alkanoylamino, thiol, alkylthio, alkylthiono, alkylsulfonyl, sulfonamido, NO2, cyano, CO2H, carbamoyl, N-hydroxycarbamoyl, N-alkylcarbamoyl, N,N-dialkylcarbamoyl, alkoxycarbonyl, (un)substituted Ph, or a combination of these groups; Y, Z = CH2, CO; R1 - R14 = H or C1-20 alkyl; R7, R8 R14 may also be aryl or aralkyl; R3, R9, R12, R13 may also be aralkyl; m, n, p = 0, 1,2; q = 0, 1-4], which effect inhibition. of farnesyl transferase, an enzyme involved in Ras oncogene expression, (no data), are prepared Any of these compds. I is used for manufacturing a medicament for treating (1) conditions requiring inhibition of prenyl transferases, farnesyl protein transferase, or tumors or (2) diseases associated with signal transaction pathways operating through Ras, proteins that are post-translationally modified by the enzyme farnesyl protein transferase, or proteins that are post-translationally modified by the enzyme geranylgeranyl protein transferase. Thus, L-methionine Me ester hydrochloride was sequentially coupled with (S)-3,4-dihydro-2,3(H)-isoquinolinedicarboxylic acid

L12 ANSWER 126 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)
2-tert-Bu ester, Boc-Val-OH, and imidazole-4-acetic acid and sapon. of the resulting tripeptide Me ester with a soln. of LiOH in H2O and HPLC purifn. to give the title compd. (II) as trifluoroacetate salt.

ACCESSION NUMBER: 1995:994541 CAPLUS
DOCUMENT NUMBER: 124:117997
TITLE: Preparation of imidazole-containing peptide and amino acid derivatives as inhibitors of farnesyl protein transferase.
INVENTOR(S): Hunt, John T.
PATENT ASSIGNEE(S): Bristol-Myers Squibb Co., USA
SOURCE: Eur. Pat. Appl., 106 pp.
CODEN: EPXXDW
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 675112	A1	19951004	EP 1995-302188	19950331
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LI, LU, MC, NL, PT, SE				
AU 9516158	A1	19951012	AU 1995-16158	19950330
HU 72440	A2	19960429	HU 1995-934	19950330
CA 2146059	AA	19951001	CA 1995-2146059	19950331
FI 9501554	A	19951001	FI 1995-1554	19950331
NO 9501266	A	19951002	NO 1995-1266	19950331
JP 07304750	A2	19951121	JP 1995-75486	19950331
CN 1112117	A	19951122	CN 1995-103978	19950331
ZA 9502696	A	19960930	ZA 1995-2696	19950331
PRIORITY APPLN. INFO.:			US 1994-221153 A	19940331
			US 1994-292916 A	19940819

OTHER SOURCE(S): MARPAT 124:117997

L12 ANSWER 127 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB The reactions of trimethylgallium and trimethylindium with a variety of secondary amines [HNMe2, HNEt2, HNPr2, HNPri2, HNBu2, HNBui2, HNBus2, HN(CH2Ph)2, HN(c-C6H11)2, HNC4H8, HNC5H10, HNC6H12 and HN(CH2CH2)2NMe], produce room-temperature stable liquid or solid adducts. These were characterized by 1H and 13C NMR, IR, mass spectrometry and elemental anal. Spectroscopic comparisons are made between these and the corresponding trimethylaluminum derivs. 1H and 13C NMR data for all three series of adducts indicate a correlation between the chemical shifts of the Me groups on the metal and the relative steric requirements of the amines. The data show a general downfield movement of these chemical shifts with increasing steric bulk.

ACCESSION NUMBER: 1995:888783 CAPLUS
DOCUMENT NUMBER: 124:87096
TITLE: Synthesis and characterization of Me3Ga and Me3In adducts of secondary amines
AUTHOR(S): Schauer, S. J.; Watkins, C. L.; Krannich, L. K.; Gala, R. B.; Gundy, E. M.; Lagrone, C. B.
CORPORATE SOURCE: Univ. of Alabama at Birmingham, Birmingham, AL, 35294, USA
SOURCE: Polyhedron (1995), 14(23/24), 3505-14
CODEN: PLYHDE; ISSN: 0277-5387
PUBLISHER: Elsevier
DOCUMENT TYPE: Journal
LANGUAGE: English

L12 ANSWER 128 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB A series of carbonaceous materials containing silicon and oxygen have been synthesized via pyrolysis of epoxy-silane composites prepared from hardened mixts. of epoxy novolac resin and epoxy-functional silane. Chemical composition of the pyrolyzed materials has been determined to be C1-y-2SizOy by a combination thermogravimetric anal., Auger electron spectroscopy, carbon, hydrogen, and nitrogen analyses, and wet chemical analyses. Pyrolysis of the epoxy novolac resin gives pure carbon made up predominantly of single graphene sheets having lateral dimension of about 20 Å which are stacked like a "house of cards.". Pyrolysis of the pure epoxy-functional silane gives C0.50Si0.19O0.31 with a glassy structure. X-ray diffraction and electrochem. tests show that pyrolyzed materials prepared from mixts. initially containing less than 50% (by weight) silane are mixts. of the carbon single-layer phase and the glassy phase, while those initially with greater than 50% silane show predominantly the glassy phase. The reversible specific capacity of these materials increases from about 500 mAh/g for the pure disordered carbon up to about 770 mAh/g in the material which contains the most silicon and oxygen. However, the voltage profile develops hysteresis of about 1 V and the irreversible capacity associated with the first reaction with lithium increases as the silicon and oxygen contents are increased. Further work is needed to eliminate these drawbacks.

ACCESSION NUMBER: 1995:820008 CAPLUS
DOCUMENT NUMBER: 123:233290
TITLE: An epoxy-silane approach to prepare anode materials for rechargeable lithium ion batteries
AUTHOR(S): Xue, J. S.; Myrtle, K.; Dahn, J. R.
CORPORATE SOURCE: Dep. of Physics, Simon Fraser Univ., Burnaby, BC, V5A 1S6, Can.
SOURCE: Journal of the Electrochemical Society (1995), 142(9), 2927-35
CODEN: JESOAN; ISSN: 0013-4651
PUBLISHER: Electrochemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English

L12 ANSWER 129 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

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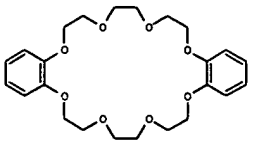

I

AB 1H NMR, mass spectra, formation consts., and crystallog. of 1:1 complexes of dibenzo crown ether I with (PhCH2)2N.HPF6 or Bu2N.HPF6 support mol. modeling calcns. of a structure in which the dialkylammonium ion is threaded through the center of I.

ACCESSION NUMBER: 1995:819794 CAPLUS
DOCUMENT NUMBER: 124:86093
TITLE: Dialkylammonium ion/crown ether complexes: the forerunners of a new family of interlocked molecules
AUTHOR(S): Ashton, Peter R.; Campbell, Paul J.; Chrystal, Ewan J. T.; Glink, Peter T.; Menzer, Stephan; Philp, Douglas; Spencer, Neil; Stoddart, J. Fraser; Tasker, Peter A.; Williams, David J.
CORPORATE SOURCE: Sch. Chem., Univ. Birmingham, Edgbaston, Birmingham, B15 2TT, UK
SOURCE: Angewandte Chemie, International Edition in English (1995), 34(17), 1865-9
CODEN: ACIEAY; ISSN: 0570-0833
PUBLISHER: VCH
DOCUMENT TYPE: Journal
LANGUAGE: English

L12 ANSWER 130 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB Porous, preferably dimensionally stable material for the removal of gaseous impurities (e.g. H2S, COS, CS2, and SO2) from gas mixture into the pores having incorporated a secondary amine which chemical bonds with the constituents to be removed. The material comprises a hydrophobic polymer with pores having an average diameter 0.1-50 μm and a secondary amine having hydrophobic properties which optionally is incorporated into a hydrophobic liquid Favorable results were attained using polypropylene as the hydrophobic polymer and ditridecyl amine as the secondary amine, with a tertiary amine, such as C12/C14-alkyl diethanol amine, being part of the hydrophobic liquid

ACCESSION NUMBER: 1995:731799 CAPLUS
DOCUMENT NUMBER: 123:117297
TITLE: Material for removal of gaseous impurities from gas mixture
INVENTOR(S): Schomaker, Elwin; Bos, Johannes
PATENT ASSIGNEE(S): Akzo Nobel N.V., Neth.
SOURCE: Eur. Pat. Appl., 9 pp.
CODEN: EPXXDW
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 662338	A1	19950712	EP 1994-203656	19941216
EP 662338	B1	20000503		
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LI, LU, MC, NL, PT, SE				
NL 9400012	A	19950801	NL 1994-12	19940106
AT 192350	E	20000515	AT 1994-203656	19941216
ES 2146635	T3	20000816	ES 1994-203656	19941216
PT 662338	T	20000929	PT 1994-203656	19941216
JP 07256096	A2	19951009	JP 1995-15567	19950106
GR 3034058	T3	20001130	GR 2000-401750	20000728
US 6355094	B1	20020312	US 2000-721017	20001122
PRIORITY APPLN. INFO.:			NL 1994-12 A	19940106
			US 1997-832331 B3	19970326

L12 ANSWER 131 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB Thermal behavior of the hexachlorozirconates of several alkanamines and aromatic mono-amines was examined using dynamic and quasi-isothermal-isobaric thermoanal. methods. Decomposition of the compds. upon an increase in temperature is accompanied by partial volatilization. The residue contains ZrO2 and is sometimes contaminated with traces of carbonization products. It is believed that the primary process, which can be summarized with the equation (ApNH4-p)2ZrCl6(s)+2aHCl(g)+2(1-a)ACl(g)+2Ap-sNH3-p+s(g)+ZrCl4(cond) (where A denotes an alkyl or aryl substituent (p = 1-4; a = 0 and s = 1 for quaternary, and a = 1 and s = 0 for other compds. studied)) is followed by instantaneous oxidation of zirconium tetrachloride remaining in the condensed phase (cond). An insight into the thermodn. of the compds. became possible on employing the van't Hoff equation to the non-isothermal thermogravimetric curves. This enabled evaluation of the enthalpies of the thermal decomposition and consequently the enthalpies of formation and the crystal lattice energies of the salts. The latter quantity was further examined using the Kapustinskii-Yatsimirskii method. Geometries, energies and other physicochem. properties of simple aliphatic and aromatic amines and their protonated forms were determined by AM1 and PM3 methods in order to reveal which of these correlate with the proton affinity of amines and the thermal behavior and thermochem. characteristics of hexachlorozirconates. In addition, the influence of dimensions of ions on the thermodn. stability of hexahalogenozirconates, with respect to dissociation and oxidation processes, was studied.

ACCESSION NUMBER: 1995:655662 CAPLUS
DOCUMENT NUMBER: 123:338901
TITLE: Thermal features and thermochemistry of hexachlorozirconates of aliphatic and aromatic mono-amines-stability of hexahalogenozirconates
AUTHOR(S): Thanh, Hoan Vu; Gruzdiewa, Ludwika; Rak, Janusz; Blazejowski, Jerzy
CORPORATE SOURCE: Department of Chemistry, University of Gdansk, Gdansk, 80-952, Pol.
SOURCE: Journal of Alloys and Compounds (1995), 224(1), 1-13
CODEN: JALCEU; ISSN: 0925-8388
PUBLISHER: Elsevier
DOCUMENT TYPE: Journal
LANGUAGE: English

L12 ANSWER 132 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB The magnitude of the γ-effects on 13C chemical shifts was studied as function of the N-substitution [Me, Et, Bu, CH2C6H5, CH2CH2C6H5, Pri, Bui, Bus, c-C6H11, CH(CH3)C6H5, But, or Ph] for several benzylamines, o-aminomethylphenols, and 3,4-dihydro-2H-1,3-benzoxazines. A correlation between the δc-values and the steric substituent consts. (E's) of the N-substituents proved useful in characterizing the variation of the γ-effects along with the conformational factors. The diastereospecificity of the γ-effects is discussed for purposes of configurational assignments.

ACCESSION NUMBER: 1995:611876 CAPLUS
DOCUMENT NUMBER: 123:82725
TITLE: Studies on the γ-effects. Part 3. Variations in the γ-effects of N-substituted benzylamines, o-aminomethylphenols and 3,4-dihydro-2H-1,3-benzoxazines against the E's substituent constants
AUTHOR(S): Neuvonen, Kari; Pihlaja, Kalevi
CORPORATE SOURCE: Department Chemistry, University Turku, Turku, Finland
SOURCE: Structural Chemistry (1995), 6(2), 77-83
CODEN: STCHES; ISSN: 1040-0400
DOCUMENT TYPE: Journal
LANGUAGE: English

L12 ANSWER 133 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB [11C]-Me chloroformate, a novel [11C]-acylating agent, was generated in situ from [11C]-methanol and phosgene. To explore the utility of [11C]-Me chloroformate, this agent was reacted with several amines to yield their corresponding [11C]-labeled Me carbamates. The average synthesis (including purification and formulation) required approx. 23 min from end of bombardment. The average specific activity was calculated to be approx. 607 mCi/μmole at end of synthesis with an average radiochem. yield of 6%, decay corrected to starting [11C]-methanol. Preliminary results reveal that [11C]-methylchloroformate is a useful general reagent for the preparation of [11C]-Me carbamates of both primary and secondary amines.

ACCESSION NUMBER: 1995:506927 CAPLUS
DOCUMENT NUMBER: 123:142977
TITLE: Synthesis of carbon-11 labeled methylcarbamates from [11C]-methylchloroformate
AUTHOR(S): Ravert, Hayden T.; Mathews, William B.; Musachio, John L.; Dannals, Robert F.
CORPORATE SOURCE: Div. Nucl. Med. Radiation Health Sci., Johns Hopkins Med. Inst., Baltimore, MD, 21205-2179, USA
SOURCE: Journal of Labelled Compounds & Radiopharmaceuticals (1995), 36(4), 365-71
CODEN: JLCRD4; ISSN: 0362-4803
PUBLISHER: Wiley
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 123:142977

L12 ANSWER 134 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB The invention provides cathepsin L inhibitors containing compds. R4-(NHCHR3CO)n-(NHCHR2CO)m-NHCHR1-X [I; R1 = H, (un)substituted arylalkyl, heterocyclic-alkyl, or lower alkyl; R2, R3 = (independently) H, (un)substituted hydrocarbyl; R4 = (un)substituted alkanoyl, sulfonyl, carbonyloxy, carbamoyl or thiocarbamoyl; X = CHO or CH2OB; B = H or OH-protecting group; m, n = (independently) 0 or 1; provided that R4 = arylalkanoyl, C>9 arylsulfonyl or lower alkylsulfonyl, or (un)substituted carbamoyl or thiocarbamoyl, when R1 = unsubstituted lower alkyl, arylalkyl, or methylthioethyl, R2 and R3 = (independently) lower alkyl or arylalkyl, X = CHO, m = 1, and n = 0 or 1] and their salts. I are useful as prophylactic/therapeutic agents for osteoporosis. For example, N-benzyloxycarbonyl-L-isoleucyl-L-tryptophanol (preparation given) was deprotected by hydrogenolysis and coupled with 1-naphthalenesulfonyl chloride in DMF containing DMAP to give 82% title alc. N-(1-naphthylsulfonyl)-L-isoleucyl-L-tryptophanol (II). Oxidation of II by pyridine-SO3 complex in DMSO gave the corresponding L-tryptophanal derivative (III), a specifically claimed compound. Human recombinant cathepsin L (preparation and purifn. given) was inhibited by III with IC50 1.9 + 10-9M. III at 10 μg/mL also gave 49% inhibition of rat bone resorption in vitro (method of Raisz). Approx. 200 I are listed with characterizing data.

ACCESSION NUMBER: 1995:435611 CAPLUS
DOCUMENT NUMBER: 122:214520
TITLE: Peptide alcohol or aldehyde derivatives as cathepsin L inhibitors and bone resorption inhibitors
INVENTOR(S): Sohda, Takashi; Fujisawa, Yukio; Yasuma, Tsuneo; Mizoguchi, Junji; Kori, Masakuni; Takizawa, Masayuki
PATENT ASSIGNEE(S): Takeda Chemical Industries, Ltd., Japan
SOURCE: Eur. Pat. Appl., 62 pp.
CODEN: EPXXDW
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 611756	A2	19940824	EP 1994-102404	19940217
EP 611756	A3	19941130		
EP 611756	B1	20030507		
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LI, LU, NL, PT, SE				
JP 07101924	A2	19950418	JP 1994-11081	19940202
JP 2848232	B2	19990120		
JP 09208545	A2	19970812	JP 1996-292418	19940202
US 5498728	A	19960312	US 1994-192038	19940204
AU 9454964	A1	19940825	AU 1994-54964	19940207
CA 2115913	AA	19940820	CA 1994-2115913	19940217
NO 9400550	A	19940822	NO 1994-550	19940217
AT 239705	E	20030515	AT 1994-102404	19940217
FI 9400788	A	19940820	FI 1994-788	19940218
HU 66219	A2	19941028	HU 1994-473	19940218
CN 1107363	A	19950830	CN 1994-101373	19940218
US 5639781	A	19970617	US 1995-495814	19950627
US 5716980	A	19980210	US 1995-495097	19950627
US 5955491	A	19990921	US 1995-495352	19950627
PRIORITY APPLN. INFO.:			JP 1993-30182	A 19930219
			JP 1993-197305	A 19930809
			JP 1994-11081	A3 19940202
			US 1994-192038	A3 19940204

L12 ANSWER 134 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)

OTHER SOURCE(S): MARPAT 122:214520

L12 ANSWER 135 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB Chiral, racemic 2-arylalkyl-2-(tetrazol-5-yl)-N-arylalkylcarboxamides were conveniently prepared from Et cyanoacetate in four steps. The synthetic methodol. developed is a facile way of introducing bulky substituents into a peptide-like framework, affording intermediate α-arylalkyl-α-amidonitriles. These nitriles were sufficiently activated to give, upon treatment with ammonium azide in DMF at 145° for twenty-four to thirty hours, the corresponding tetrazoles in good yields. It has been determined that an optically pure α-arylalkyl-α-amidonitrile epimerized to give diastereomeric products under the above conditions. A procedure for the fractional crystallization of the (S)-(-)-α-methylbenzylamine salts of the tetrazoles to give the optically enriched tetrazoles was also developed.

ACCESSION NUMBER: 1995:389451 CAPLUS
DOCUMENT NUMBER: 123:169560
TITLE: Synthesis and resolution of 2-arylalkyl-2-(tetrazol-5-yl)-N-arylalkylcarboxamides. A new class of chiral sterically hindered tetrazole derivatives
AUTHOR(S): Moriarty, Robert M.; Levy, Stuart G.
CORPORATE SOURCE: Dep. Chem., Univ. Illinois, Chicago, IL, 60680, USA
SOURCE: Journal of Heterocyclic Chemistry (1995), 32(1), 155-60
CODEN: JHTCAD; ISSN: 0022-152X
PUBLISHER: HeteroCorporation
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 123:169560

L12 ANSWER 136 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB Dibenzylamido anions ((PhCH2)2N-) can be transformed into 1,3-diphenyl-2-azaallyl anions ((PhC(H)⁻⁻-NCPh)-) by the assistance of PMDETA- ((Me2NCH2CH2)2NMe) complexed Li+, Na+, or K+ cations. The heavier alkali-metal cations give only the trans,trans conformation of the azaallyl anion, in contrast to the lighter Li+ cation, which yields two crystalline conformers, the trans,trans and an unknown species. Ab initio MO geometry optimizations on model Li and Na complexes intimate that it is the relative tightness of the contact ion pair structures which dictates this distinction with Li+ having more influence on the conformation and stability of the anion than Na+, which forms a much looser contact ion pair more akin to the free anion. On the basis of kinetic 1H NMR studies, combined with x-ray crystallog. data, the amido → azaallyl conversion can be explained in terms of a two-step process involving β-elimination of a metal hydride followed by hydride metalation of the produced imine PhCHZN:C(H)Ph. This process appears to be initiated by deaggregation of the metallodibenzylamine to an intermediate monomeric structure, accomplished by solvation. The nature and degree of solvation required depend on the particular M+ cation involved. Three new crystal structures are revealed in the course of this study. All are based on familiar four-membered (N-M)2 rings, but whereas the sodium complex {{(PhCH2)2NNa·TMEDA}2} and the lithium complex {{(PhCH2)2NLi·THF}2} are both discrete dimers, unique [{[(PhCH2)2NLi]2·(dioxane)}∞], isolated as its toluene hemisolvate, is a polymer composed of linked dimeric units and so is the first dibenzylamido alkali-metal species to have an infinitely extended structure.

ACCESSION NUMBER: 1995:283571 CAPLUS
DOCUMENT NUMBER: 122:187642
TITLE: Synthetic, Structural, Mechanistic, and Theoretical MO Studies of the Alkali-Metal Chemistry of Dibenzylamine and Its Transformation to 1,3-Diphenyl-2-azaallyl Derivatives
AUTHOR(S): Andrews, Philip C.; Armstrong, David R.; Baker, Daniel R.; Mulvey, Robert E.; Clegg, William; Horsburgh, Lynne; O'Neil, Paul A.; Reed, David
CORPORATE SOURCE: Department of Pure and Applied Chemistry, University of Strathclyde, Glasgow, G1 1XL, UK
SOURCE: Organometallics (1995), 14(1), 427-39
CODEN: ORGND7; ISSN: 0276-7333
PUBLISHER: American Chemical Society
DOCUMENT TYPE: Journal
LANGUAGE: English

L12 ANSWER 137 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB (S)-Me2CHCH(OH)CH2NH2 (I) was prepared from D-valine (II) in a multistep synthesis. Thus, known conversion of II to (S)-1,2-epoxy-3-methylbutane was followed by ring opening with (PhCH2)2NLi at -78° to give (S)-Me2CHCH(OH)CH2N(CH2Ph)2 which was hydrogenolized to I. The enantiomeric purity of I (97.2 ± 0.2% ee) is determined by GC of the oxazolidin-2-one derivative on both L- and D-Chirasil-Val. The procedure
provides a useful route to both enantiomers of 1-amino-2-alkanols starting from L- and D-amino acids, resp.

ACCESSION NUMBER: 1995:228185 CAPLUS
DOCUMENT NUMBER: 122:105205
TITLE: A useful route to both enantiomers of 1-amino-2-alkanols: synthesis of 1-amino-3-methyl-2-butanol from valine
AUTHOR(S): Koppenhoefer, Bernhard; Trettin, Ulrich; Waechtler, Andreas
CORPORATE SOURCE: Institut fuer Organische Chemie, Univ. Tuebingen, Tuebingen, D-72076, Germany
SOURCE: Synthesis (1994), (11), 1141-2
CODEN: SYNTBF; ISSN: 0039-7881
PUBLISHER: Thieme
DOCUMENT TYPE: Journal
LANGUAGE: English

L12 ANSWER 138 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

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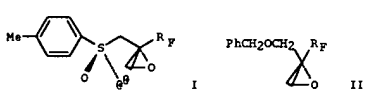

I II

AB New optically pure poly-halo and poly-fluoro oxiranes I (2S,RS-isomers) {RF = CH2F, CF2H, CF2Cl, CF3, CF2CF3, (CF2)6CF3} and the 2R,RS isomers were synthesized by addition of diazomethane on the corresponding β-keto-γ-fluoro substituted sulfoxide intermediates, which are in keto, hydrate, or keto/hydrate forms. Syntheses of sulfur-free fluorinated oxiranes II, (S)-HOCMe(CF3)CH2N(CH2Ph)2, acids (R)-HO2CC(OH)(CF3)CH2R1 {R1 = (PhCH2)2N, PhCH2O}, and diols (R)-HOCH2C(OH)(CF2X)CH2N(CH2Ph)2 (X = F, Cl) are examples of the chemical versatility of the oxiranes.

ACCESSION NUMBER: 1995:30146 CAPLUS
DOCUMENT NUMBER: 123:168931
TITLE: New fluorinated chiral synthons
AUTHOR(S): Bravo, Pierfrancesco; Farina, Alessandra; Frigerio, Massimo; Meille, Stefano Valdo; Viani, Fiorenza; Soloshonok, Vadim
CORPORATE SOURCE: Dipartimento di Chimica, Politecnico di Milano, Milan, I-20131, Italy
SOURCE: Tetrahedron: Asymmetry (1994), 5(6), 987-1004
CODEN: TASYE3; ISSN: 0957-4166
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 123:168931

L12 ANSWER 139 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB The thermal decomposition of zinc dibenzyldithiocarbamate (ZnDBzDTC), a compound used in the formulation of rubber and a possible precursor for N-nitrosodibenzylamine (NDBzA), was studied by a variety of thermal and spectroscopic techniques. At 326°C, the decomposition temperature of the dithiocarbamate, carbon disulfide and dibenzylamine were the principal products formed. Smaller amts. of toluene, benzyl isothiocyanate, N,N,N'-tribenzylthiourea, and benzylbenzylidene were identified. The amount of dibenzylamine (DBzA) formed by the thermal decomposition of ZnDBzDTC may have a limited role in the formation of NDBzA in hams processed in elastic rubber nettings. The thermal conditions used in the smokehouse are significantly lower than the decomposition temperature of purified ZnDBzDTC.

ACCESSION NUMBER: 1994:654180 CAPLUS
DOCUMENT NUMBER: 121:254180
TITLE: Thermal decomposition of the rubber vulcanization agent, zinc dibenzyldithiocarbamate, and its potential role in nitrosamine formation in hams processed in elastic nettings
AUTHOR(S): Helmick, John S.; Fiddler, Walter
CORPORATE SOURCE: Eastern Regional Research Center, U.S. Department of Agriculture, Philadelphia, PA, 19118, USA
SOURCE: Journal of Agricultural and Food Chemistry (1994), 42(11), 2541-4
CODEN: JAFCAU; ISSN: 0021-8561
DOCUMENT TYPE: Journal
LANGUAGE: English

L12 ANSWER 140 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB The com. epoxidized and with butanol esterified soybean and sunflower oil was by means, of epoxy group chemical modified with low-mol. compds. having an amine hydrogen. Epoxidized butanol esters of soybean and sunflower oil mixts. were reacted with amines. The conditions of the reactions, their catalysis, and their rate consts. were determined Useful nonvolatile additives for polymers were prepared by reactions with certain functionalized amines. The mol. weight of the additives could be increased by converting them to Ca salts. The modified oil is thermally more stable than the original oil.

ACCESSION NUMBER: 1994:324870 CAPLUS
DOCUMENT NUMBER: 120:324870
TITLE: Modified soybean oil as a nonvolatile additive for polymers I. Amines bonded on oil
AUTHOR(S): Citovicky, P.; Sedlar, J.; Chrastova, V.; Ondas, M.
CORPORATE SOURCE: Fac. Chem. Technol., Slovak Tech. Univ., Bratislava, SK-812 37, Slovakia
SOURCE: Chemical Papers (1993), 47(5), 325-30
CODEN: CHPAEG; ISSN: 0366-6352
DOCUMENT TYPE: Journal
LANGUAGE: English

L12 ANSWER 141 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB In the title separation method, octahydrophenazine-containing caprolactam is mixed with one or more amines selected from secondary amines having b.ps. 280 - 350° and primary amines having ether bonds and is then distilled Said primary amines have b.ps. 230 -350°.

ACCESSION NUMBER: 1994:299524 CAPLUS
DOCUMENT NUMBER: 120:299524
TITLE: Separation of octahydrophenazine from caprolactam
INVENTOR(S): Too, Yasuhiko; Sugita, Keisuke; Kajikuri, Koji
PATENT ASSIGNEE(S): Sumitomo Chemical Co., Ltd., Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 3 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 06072998	A2	19940315	JP 1992-227064	19920826
JP 3254744	B2	20020212		
PRIORITY APPLN. INFO.:			JP 1992-227064	19920826

L12 ANSWER 142 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB R1R2NCH2CH:CHCH2NR3R4 (I; R1-3 = C8-30 alkyl, C1-20 substituted with C5-12 cycloalkyl; R1-4 C5-12 cycloaalkyl, c3-20 alkenyl), useful as effective antioxidant protectant for lubricants and/or synthetic polymers, are prepared Ph2NH and AcOCH2CH:CHCH2OAc in THF was treated with (Ph3P)4Pd to give 1-acetoxy-4-benzylamino-2-butene which with dicyclohexylmethylamine in THF was treated with (Ph3P)4Pd to give I (R1 = R2 = cyclohexylmethyl, R3 = R4 = PhCH2). Similarly prepared was I (R1 = R2 = Ph, R3 = R4 = PhCH2) (II). In a process stabilization of dynamically Geolast II, showed 77% retention of elongation after 7 days at 135°.

ACCESSION NUMBER: 1994:216703 CAPLUS
DOCUMENT NUMBER: 120:216703
TITLE: Preparation of substituted 1,4-diamino-2-butene stabilizers
INVENTOR(S): Babiarz, Joseph E.; Cunkle, Glen T.; Rutsch, Werner
PATENT ASSIGNEE(S): Ciba-Geigy Corp., USA
SOURCE: U.S., 12 pp. Cont.-in-part of U.S. Ser. No. 400,649, abandoned.
CODEN: USXXAM
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 3
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 5283367	A	19940201	US 1991-701268	19910516
ES 2050413	T3	19940516	ES 1990-810636	19900822
JP 03093751	A2	19910418	JP 1990-229510	19900830
EP 514333	A2	19921119	EP 1992-810337	19920507
EP 514333	A3	19930512		
R: BE, DE, ES, FR, GB, IT, NL				
CA 2068661	AA	19921117	CA 1992-2068661	19920514
JP 05186770	A2	19930727	JP 1992-148728	19920515
US 5391808	A	19950221	US 1993-146377	19931101
US 5492954	A	19960220	US 1994-341719	19941118
PRIORITY APPLN. INFO.:			US 1989-400649 B2	19890830
			US 1991-701268 A	19910516
			US 1993-146377 A3	19931101
OTHER SOURCE(S):	MARPAT 120:216703			

L12 ANSWER 143 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
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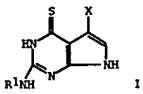

I

AB The title compds. I (R1 = acyl; X = CH2NR2R3; R2, R3 = primary alkyl, alkenyl, or aralkyl; R2R3 may form ring) or their salts are prepared by reaction of I (X = H) with R2R3NH (R2, R3 = same as I). A mixture of 3.5 g I (R1 = n-octanoyl, X = H) and 9.5 g dibenzylamine in H2O-AcOH was treated with formalin at 60° for 14 h and treated with HCl-MeOH at 60° for 1.5 h to give 3.95 g I [R1 = n-octanoyl, X = CH2N(CH2Ph)2] (II). II was converted into I [R1 = H, X = (3S,4R,5S)-4,5-dihydroxycyclopent-1-en-3-ylaminomethyl], which had IC50 of 22 μg/mL in vitro against mouse tumor cells.

ACCESSION NUMBER: 1994:164217 CAPLUS
DOCUMENT NUMBER: 120:164217
TITLE: 6-Thio-7-deazapurines as intermediates for antitumor agents and microbicides and their preparation
INVENTOR(S): Nishimura, Susumu; Nomura, Masaaki
PATENT ASSIGNEE(S): Takeda Chemical Industries Ltd., Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 8 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 05230064	A2	19930907	JP 1991-182358	19910723
JP 07100706	B4	19951101		
PRIORITY APPLN. INFO.:			JP 1991-182358	19910723

OTHER SOURCE(S): MARPAT 120:164217

L12 ANSWER 144 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
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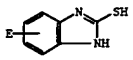

I

AB Blends of N,N,N',N'-tetrasubstituted 1,4-diamino-2-butene (alkyl, cycloalkyl, aralkyl, aryl or mixture as substituents) and mercaptoimidazole I (E = H, alkyl, cycloalkyl, aryl or phenylalkyl) are claimed. A 50:50 blend of N,N,N',N'-tetradecyl-2-butene-1,4-diamine and 2-mercaptotolylimidazole was added at 2% in crosslinked polypropylene/nitrile rubber to give a product vulcanizate having elongation 81% (retention after 7 days at 135°).

ACCESSION NUMBER: 1994:136824 CAPLUS
DOCUMENT NUMBER: 120:136824
TITLE: N,N'-alkenylene amine/mercaptotolylimidazole blends as high temperature antioxidants for elastomers
INVENTOR(S): Horsey, Douglas W.; Patel, Ambelal R.
PATENT ASSIGNEE(S): Ciba-Geigy Corp., USA
SOURCE: U.S., 10 pp.
CODEN: USXXAM
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 5240976	A	19930831	US 1992-934092	19920821
EP 585202	A1	19940302	EP 1993-810574	19930812
R: DE, FR, GB, IT				
JP 06184361	A2	19940705	JP 1993-225257	19930818
CA 2104408	AA	19940222	CA 1993-2104408	19930819
PRIORITY APPLN. INFO.:			US 1992-934092 A	19920821

L12 ANSWER 145 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
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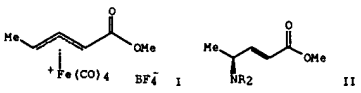

I II

AB The nucleophilic addition of nitrogen nucleophiles, R2NH (e.g., R = PhCH2), to the highly enantiomerically enriched iron complex I (ee ≥ 95%) leads, after oxidative removal of the Fe(CO)4 group, to 4-amino-enoates (S)-II of high enantiomeric purity (ee = 95-98%). The reaction is highly regio- and stereoselective and proceeds in good yields without isomerization of the double bond.

ACCESSION NUMBER: 1994:8196 CAPLUS
DOCUMENT NUMBER: 120:8196
TITLE: Iron mediated synthesis of 4-amino-enoates of high enantiomeric purity
AUTHOR(S): Enders, Dieter; Finkam Michael
CORPORATE SOURCE: Inst. Org. Chem., Rheinisch-Westfael. Tech. Hochsch., Aachen, D-5100, Germany
SOURCE: Synlett (1993), (6), 401-2
CODEN: SYNLES; ISSN: 0936-5214
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 120:8196

L12 ANSWER 146 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB The compds., tetrasubstituted with alkyl, aralkyl, or aryl groups, are useful as antioxidants or heat stabilizers for synthetic polymers or lubricants. N,N,N',N'-tetradecyl-2-butene-1,4-diamine was prepared and used as a stabilizer for Geolast (a crosslinked polypropylene-nitrile rubber resin).

ACCESSION NUMBER: 1993:582056 CAPLUS
DOCUMENT NUMBER: 119:182056
TITLE: Substituted 1,4-diamino-2-butene stabilizers and stabilized compositions
INVENTOR(S): Babiarz, Joseph E.; Cunkle, Glen T.; Rutsch, Werner
PATENT ASSIGNEE(S): Ciba-Geigy A.-G., Switz.
SOURCE: Eur. Pat. Appl., 24 pp.
CODEN: EPXXDW
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 3
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 514333	A2	19921119	EP 1992-810337	19920507
EP 514333	A3	19930512		
R: BE, DE, ES, FR, GB, IT, NL				
US 5283367	A	19940201	US 1991-701268	19910516
PRIORITY APPLN. INFO.:			US 1991-701268 A	19910516
			US 1989-400649 B2	19890830

OTHER SOURCE(S): MARPAT 119:182056

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AB The reactions of (Me3Al)2 with 11 aminoarsines, Me2AsR [R = Et2N, Pr2N, (Me2CH)2N, Bu2N, (Me2CHCH2)2N, C4H8N, C5H10N, C6H12N, CH3NC4H8N, Ph2N, (PhCH2)2N] were studied by multinuclear NMR spectroscopy. The results are compared with those of the authors' previous studies on the Me3Al/Me2AsNMe2 system. In each case, except Me2AsNPh2, the final reaction products are [Me2AlR]2 and Me3As. The reaction intermediates were identified and, in most cases, the As-N-Al adducts and Me2AlR·AlMe3 are observed With Me2AsNPh2 the product is Me3As·Me2AlNPh2. The influence of steric and electronic effects on arsenic vs. nitrogen bonding site preference, adduct stability, complexity of overall reaction and ease of forming Me3As and [Me2AlR]2 are discussed. [Me2AlR]2, Me2AlR·AlMe3 and Me3Al·HR were independently synthesized and characterized. A comparison of the 13C NMR chemical shift values for Me2AsR and Me2AsR·AlMe3 provides information on steric interactions that influence adduct stability.

ACCESSION NUMBER: 1993:428193 CAPLUS
DOCUMENT NUMBER: 119:28193
TITLE: Reactivity of bis(trimethylaluminum) with selected aminoarsines and secondary amines
AUTHOR(S): Thomas, C. J.; Krannich, L. K.; Watkins, C. L.
CORPORATE SOURCE: Dep. Chem., Univ. Alabama, Birmingham, AL, 35294, USA
SOURCE: Polyhedron (1993), 12(4), 389-99
CODEN: PLYHDE; ISSN: 0277-5387
DOCUMENT TYPE: Journal
LANGUAGE: English

L12 ANSWER 148 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

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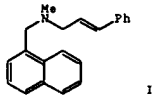

I

AB Reaction of vinyl boronic acids with the adducts of secondary amines and paraformaldehyde gives tertiary allylamines with the same geometry. This simple and practical method was used for the synthesis of geometrically pure naftifine (I), a potent antifungal agent. Thus, condensation of (CH2O)n with 1-(N-methylaminomethyl)naphthalene afforded a hydroxymethylamine derivative which was reacted with (E)-PhCH:CHB(OH)2 to afford I in 82% yield.

ACCESSION NUMBER: 1993:233548 CAPLUS
DOCUMENT NUMBER: 118:233548
TITLE: The boronic acid Mannich reaction: a new method for the synthesis of geometrically pure allylamines
AUTHOR(S): Petasis, Nicos A.; Akritopoulou, Irini
CORPORATE SOURCE: Dep. Chem., Univ. South. California, Los Angeles, CA, 90089-0744, USA
SOURCE: Tetrahedron Letters (1993), 34(4), 583-6
CODEN: TELEAY; ISSN: 0040-4039
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 118:233548

L12 ANSWER 149 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

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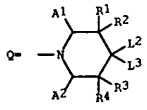

AB RCH2CH:CHCH2R [I; R = piperidino group Q; 1 R may be bis(substituted C1-30 alkyl)amino; A1, A2 = (substituted) aryl; L2 = H, OH, alkoxy, alkanoyloxy, etc. and L2 = H; L2, L3 = OH, alkoxy, alkylamino, etc.; L2L3 = O; R1-R4 = H, (substituted) C1-30 alkyl], useful as antioxidants for synthetic polymers and rubbers (no data), were prepared Thus, AcOCH2CH:CHCH2OAc was condensed with 2,6-diphenylpiperidine to give I (R = 2,6-diphenylpiperidino).

ACCESSION NUMBER: 1993:212899 CAPLUS
DOCUMENT NUMBER: 118:212899
TITLE: Preparation of 1,4-bis(2,6-diarylpiperidino)-2-butene and analogs as antioxidants and light and heat stabilizers
INVENTOR(S): Cunkle, Glen T.; Babiarz, Joseph E.
PATENT ASSIGNEE(S): Ciba-Geigy A.-G., Switz.
SOURCE: Eur. Pat. Appl., 18 pp.
CODEN: EPXXDW
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 521820	A1	19930107	EP 1992-810399	19920526
EP 521820	B1	19960207		
R: BE, DE, ES, FR, GB, IT, NL				
US 5204474	A	19930420	US 1991-709688	19910603
CA 2070121	AA	19921204	CA 1992-2070121	19920601
JP 05194388	A2	19930803	JP 1992-168647	19920603
US 5290940	A	19940301	US 1992-990215	19921214
PRIORITY APPLN. INFO.:			US 1991-709688 A	19910603
OTHER SOURCE(S):	MARPAT 118:212899			

L12 ANSWER 150 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

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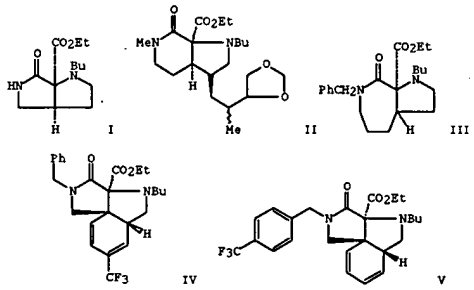

AB The scope and limitations of the intramol. 1,3-dipolar cycloaddn. of doubly-stabilized azomethine ylides to unactivated olefinic, acetylenic, and aromatic dipolarophiles was studied. The azomethine ylides studied were generated by flash vacuum pyrolysis of their corresponding aziridines and were found to add stereospecifically in good to excellent yields to a variety of unactivated dipolarophiles. Generation of the diazabicyclo[3.3.0]octane (e.g., I), diazabicyclo[4.3.0]nonane (e.g., II), and diazabicyclo[5.3.0]decane (e.g., III) ring systems are possible using this technol. In addition, the first examples of cycloaddn. of a stabilized azomethine ylide to benzene dipolarophiles are reported. Cycloaddns. of this type generate highly functionalized tricyclic systems with complete relative stereocontrol at the newly formed stereocenters. Cycloadducts IV and V are in equilibrium, presumably by way of the intermediate azomethine ylide, under conditions of flash vacuum pyrolysis.

ACCESSION NUMBER: 1993:38786 CAPLUS
DOCUMENT NUMBER: 118:38786
TITLE: Intramolecular 1,3-dipolar cycloaddition of stabilized azomethine ylides to unactivated dipolarophiles
AUTHOR(S): Henke, Brad R.; Kouklis, Andrew J.; Heathcock, Clayton H.
CORPORATE SOURCE: Dep. Chem., Univ. California, Berkeley, CA, 94720, USA
SOURCE: Journal of Organic Chemistry (1992), 57(26), 7056-66
CODEN: JOCEAH; ISSN: 0022-3263
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 118:38786

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AB A polymer composition (e.g., a polyolefin or synthetic elastomer) is stabilized against heat and O with I of the title compds. Antioxidant effectiveness of 0.5 wt% tetraphenyl-2-butyne-1,4-diamine (I) in 10W30 engine oil by ASTM Method D4742 gave oxidation induction time 237 min. vs. 113 min. for a control containing no I.

ACCESSION NUMBER: 1993:23257 CAPLUS
DOCUMENT NUMBER: 118:23257
TITLE: N,N,N',N'-Tetrasubstituted 1,4-diamino-2-butyne or N,N-disubstituted propargylamine as stabilizers for polymer compositions
INVENTOR(S): Babiarz, Joseph E.; Rutsch, Werner
PATENT ASSIGNEE(S): Ciba-Geigy Corp., USA
SOURCE: U.S., 11 pp.
CODEN: USXXAM
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 5151459	A	19920929	US 1991-701267	19910516
PRIORITY APPLN. INFO.:			US 1991-701267	19910516

OTHER SOURCE(S): MARPAT 118:23257

L12 ANSWER 152 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

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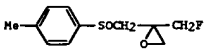

I

AB Optically pure (2S,Rs)-2-(fluoromethyl)-2-[(4-methylphenylsulfinyl)methyl]oxirane (I) was obtained in good yield in high diastereomeric excess by reacting diazomethane with optically pure 1-fluoro-3-(4-methylphenylsulfinyl)-2-propanone. Regio- and stereoselective openings of the oxirane ring of I with selected nucleophiles afforded a number of useful derivs.

ACCESSION NUMBER: 1993:6802 CAPLUS
DOCUMENT NUMBER: 118:6802
TITLE: A new versatile fluorinated C4 chiron
AUTHOR(S): Arnone, Alberto; Bravo, Pierfrancesco; Cavicchio, Giancarlo; Frigerio, Massimo; Marchetti, Valeria; Viani, Fiorenza; Zappala, Carmela
CORPORATE SOURCE: Cent. Stud. Sostanze Org. Nat., CNR, Milan, I-20133, Italy
SOURCE: Tetrahedron Letters (1992), 33(38), 5609-12
CODEN: TELEAY; ISSN: 0040-4039
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 118:6802

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AB Treatment of carbamate (PhCH2)2NCH2CH2OCby (Cby = 1,3-oxazolidin-3-ylcarbonyl) with sec-BuLi and (-)-sparteine in Et2O at -78°, followed by reaction with CO2-CH2N2 and reduction with LiAlH4 gave (R)-(PhCH2)2NCH2CH2CH(OH)CH2OH. MeI, Me3SiCl, Bu3SnCl, and Me2CHCHO were also used as electrophiles. (S)-N,N-Dibenzylleucinol or (S)-N,N-dibenzylprolinol carbamates were reacted similarly.

ACCESSION NUMBER: 1993:6599 CAPLUS
DOCUMENT NUMBER: 118:6599
TITLE: Stereoselective generation of 1-alkoxy-2-amino carbanions via deprotonation. Synthesis of enantiomerically and diastereomerically pure β-amino alcohols
AUTHOR(S): Schwerdtfeger, Joerg; Hoppe, Dieter
CORPORATE SOURCE: Inst. Org. Chem., Univ. Kiel, Kiel, W-2300, Germany
SOURCE: Angewandte Chemie (1992), 104(11), 1547-9 (See also Angew. Chem., Int. Ed. Engl., 1992, 31(11), 1505-7)
CODEN: ANCEAD; ISSN: 0044-8249
DOCUMENT TYPE: Journal
LANGUAGE: German
OTHER SOURCE(S): CASREACT 118:6599

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AB Reduction of Et benzoate by the title aluminate and by related compds. was investigated. Replacement of the piperidino group by bulky or less nucleophilic amino groups decreased the yield of PhCHO drastically. The mechanism involves formation of two unstable intermediates by the attack of hydride or piperidino groups on the sp2 C of the ester, followed by their conversion into a more stable intermediate, an α-piperidino alkoxoaluminate.

ACCESSION NUMBER: 1992:530705 CAPLUS
DOCUMENT NUMBER: 117:130705
TITLE: Mechanism of aldehyde synthesis from ester by sodium diethylpiperidinohydroaluminate
AUTHOR(S): Yoon, Nung Min; Ahn, Jin Hee; An, Duk Keun
CORPORATE SOURCE: Dep. Chem., Sogang Univ., Seoul, 121-742, S. Korea
SOURCE: Bulletin of the Korean Chemical Society (1992), 13(3), 339-41
CODEN: BKCSDE; ISSN: 0253-2964
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 117:130705

L12 ANSWER 155 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
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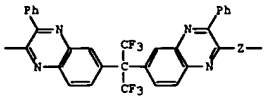

I

AB The title polymers have repeating unit I (Z = 1,3- or 1,4-phenylene) and good thermal stability, and are useful as dielecs. in elec. apparatus I (Z = 1,4-phenylene) had glass temperature 300°, thermal decomposition threshold (in air) 450°, and dielec. constant 2.8.

ACCESSION NUMBER: 1992:256298 CAPLUS
DOCUMENT NUMBER: 116:256298
TITLE: Fluorine-containing polyquinoxalines, their preparation from fluorine-containing aromatic tetraamines and their applications
INVENTOR(S): Garapon, Jacques; Bardon, Genevieve; Sillion, Bernard
PATENT ASSIGNEE(S): Institut Francais du Petrole, Fr.
SOURCE: Fr. Demande, 20 pp.
CODEN: FRXXBL
DOCUMENT TYPE: Patent
LANGUAGE: French
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
FR 2661679	A1	19911108	FR 1990-5623	19900502
FR 2661679	B1	19920814		
JP 04227721	A2	19920817	JP 1991-100103	19910501
JP 2969482	B2	19991102		
PRIORITY APPLN. INFO.:			FR 1990-5623 A	19900502

L12 ANSWER 156 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB Strong bases added to the mobile phase dramatically improve the peak shapes of phenylenediamines and benzylamines. Acidic ion-pairing additives do not improve peak shapes, suggesting peak improvement involves ion suppression. The solutes produce very poor peak shapes or do not elute using pure or methanol-modified supercrit. fluids from either standard or deactivated columns. Decreasing the stationary phase polarity and improving deactivation are ineffective alone in improving peak shapes.

ACCESSION NUMBER: 1991:573815 CAPLUS
DOCUMENT NUMBER: 115:173815
TITLE: Effect of basic additives on peak shapes of strong bases separated by packed-column supercritical fluid chromatography
AUTHOR(S): Berger, Terry A.; Deye, Jerome F.
CORPORATE SOURCE: Hewlett-Packard, Co., Avondale, PA, 19311-0900, USA
SOURCE: Journal of Chromatographic Science (1991), 29(7), 310-17
CODEN: JCHSBZ; ISSN: 0021-9665
DOCUMENT TYPE: Journal
LANGUAGE: English

L12 ANSWER 157 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB The monooxygenase and oxidase activities of liver microsomes from phenobarbital (PB)-treated rabbits were investigated for their dependence on the high spin shift (Δα) of the ferric cytochrome P 450 induced by a series of benzphetamine analogs. The spin shift activity of the substrate dets., via the 1st electron transfer kinetics, the steady-state level of the reaction intermediate oxycytochrome P 450. Correlation of the amount or oxycytochrome P 450 with Δα can be exptl. proved. The spin-state-dependent formation of oxycytochrome P 450 regulates quant. the rates of NADPH oxidation and substrate N-demethylation. Both activities correlate with Δα. Oxycytochrome P 450 is substrate-stabilized toward decay with the formation of O2- which, upon dismutation, gives rise to H2O2. The ratio of N-demethylase to NADPH oxidase activity (coupling ratio) also increases with the spin shift, Δα. Concomitantly, the proportion of NADPH accounted for by H2O2 and H2O formation via 2- and 4-electron reduction of O2 decreases.
This indicates that the substrate-induced structural changes in the enzyme active center which give rise to spin transition may likewise modify the coupling properties. Perfluorinated compds., which fail to undergo monooxygenation, fall in line with the benzphetamine derivs. with respect to the dependence of NADPH oxidation rate and steady-state oxycytochrome P 450 level on Δα. The increased oxidase activity results mostly in H2O formation. The leakiness of the PB-induced monooxygenase pathway in the biotransformation of O2 in the presence of the benzphetamines and perfluorinated compds. does not result in marked increases in H2O2 formation. Therefore, the increase of NADPH oxidase activity by these substrates does not significantly enhance H2O2-mediated O2 tissue toxicity.

ACCESSION NUMBER: 1991:202298 CAPLUS
DOCUMENT NUMBER: 114:202298
TITLE: Cytochrome P-450 spin state and leakiness of the monooxygenase pathway
AUTHOR(S): Blanck, J.; Ristau, O.; Zhukov, A. A.; Archakov, A. I.; Rein, H.; Ruckpaul, K.
CORPORATE SOURCE: Cent. Inst. Mol. Biol., Acad. Sci. GDR, Berlin, 11115, Ger. Dem. Rep.
SOURCE: Xenobiotica (1991), 21(1), 121-35
CODEN: XENOEH; ISSN: 0049-8254
DOCUMENT TYPE: Journal
LANGUAGE: English

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AB The reaction between Pd(dmpe)Me2, [dmpe = 1,2-bis(dimethylphosphino)ethane] and [HNRR'R'']X {[NH4]PF6, [NH4]BPh4, [NH3Et]BPh4, [NH2Et2]BF4, [NH2Et2]BPh4, [NHEt3]BP4, [NH2-i-Pr2]BPh4, and [1-methylimidazolium]BPh4] in CH2Cl2 or CH3CN rapidly produces CH4 and the corresponding amine complexes [Pd(dmpe)Me(NRR'R'')]X in 57-87% yield. Cone angles for these and other amines were determined from geometric measurements of CPK models. Equilibrium binding consts. for 16 amine ligands to the Pd(dmpe)Me+ Lewis acid were measured by variable-temperature 31P NMR spectroscopy. Of the various amine ligands studied, 1-methylimidazole and ethylamine bind most effectively. This parallels the role of histidine and lysine for binding metals in metalloproteins.

ACCESSION NUMBER: 1991:143670 CAPLUS
DOCUMENT NUMBER: 114:143670
TITLE: Cone angles for amine ligands. X-ray crystal structures and equilibrium measurements for ammonia, ethylamine, diethylamine, and triethylamine complexes with the [bis(dimethylphosphino)ethane]methylpalladium (II) cation
AUTHOR(S): Seligson, Allen L.; Trogler, William C.
CORPORATE SOURCE: Dep. Chem., Univ. California, La Jolla, CA, 92093-0506, USA
SOURCE: Journal of the American Chemical Society (1991), 113(7), 2520-7
CODEN: JACSAT; ISSN: 0002-7863
DOCUMENT TYPE: Journal
LANGUAGE: English

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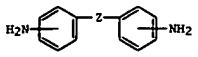

I

AB Pos.-working polyamic acid photoresist compns. are described having improved high resolution upon image development and exhibiting stable photosensitivity and superior dielec. performance. The compns. are comprised of the condensation product of an aromatic dianhydride and an aromatic diprimary amine containing 10-50 mol.% of the primary diamine I (Z = O, SO2, alkylene, fluoroalkylene, or biphenylylene) and a diazoquinone photoactive sensitizer. The composition can be prebaked at ≥120° prior to development without degradation of its photosensitivity and development. Thus, a solution containing a 3,3',4,4'-benzophenonetetracarboxylic acid dianhydride-4-aminophenyl sulfone-4-(4-aminophenoxy)phenyl sulfone copolymer and a diazoquinone photosensitizer was overcoated on a treated Si wafer, baked, exposed through a photomask to a Hg lamp, and developed with Shipley MF-312 to resolve 5 μm lines and spaces.

ACCESSION NUMBER: 1991:72341 CAPLUS
DOCUMENT NUMBER: 114:72341
TITLE: Positive-working polyamic acid/imide photoresist compositions and their use as dielectrics
INVENTOR(S): Brewer, Terry; Moss, Mary; Cuzmar, Ruth; Hawley, Dan; Flaim, Tony
PATENT ASSIGNEE(S): Brewer Science, Inc., USA
SOURCE: PCT Int. Appl., 27 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.		DATE
WO 9005382	A1	19900517	WO 1989-US4976		19891107
W: AU, JP, KR					
RW: AT, BE, CH, DE, FR, GB, IT, LU, NL, SE					
US 5024922	A	19910618	US 1988-268023		19881107
AU 8946461	A1	19900528	AU 1989-46461		19891107
PRIORITY APPLN. INFO.:			US 1988-268023	A	19881107
			WO 1989-US4976	A	19891107

L12 ANSWER 160 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
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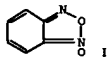

I

AB ESR study of photolysis of 2,1,3-benzoxadiazole 1-oxide (I) in the presence of R1R2NH (R1 = Ph, PhCH2, Et, Me2CH; R2 = Ph, Me, PhCH2 Me2CH, Et) showed that R1R2NO• radicals were the stable products, through an oxygen-transfer exciplex and N-H bond cleavage.

ACCESSION NUMBER: 1991:23311 CAPLUS
DOCUMENT NUMBER: 114:23311
TITLE: ESR study of photochemical reaction of 2,1,3-benzoxadiazole-1-oxide with secondary amines
AUTHOR(S): Feng, Liangbo; Wang, Hanqing
CORPORATE SOURCE: Lanzhou Inst. Chem. Phys., Chin. Acad. Sci., Lanzhou, Peop. Rep. China
SOURCE: Bopuxue Zazhi (1990), 7(2), 187-94
CODEN: BOZAE2; ISSN: 1000-4556
DOCUMENT TYPE: Journal
LANGUAGE: Chinese

L12 ANSWER 161 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB An extensive series of N-(monoethylphosphoryl) peptides was synthesized and their inhibition of purified human skin fibroblast collagenase examined. At the cleavage site S1, all reported compds. have the (EtO)(OK)P(O) group and the peptide side chain extended toward the C-terminal end (up to P5') of the substrate sequence. These phorphoramidates with a tetrahedrally hybridized P atom are thought to be transition state analog inhibitors. They exhibited fair inhibitory potency against this vertebrate collagenase. The most potent of these, (EtO)(OK)P(O)-Ile-Trp-NHMe, is nearly 100 times stronger than (EtO)(OK)P(O)-Ile-Ala-Gly-OK (I), which has the sequence matching that of the α1(I) chain of collagen in P1', P2', P3' after the cleavage site. Several compds. were prepared in an attempt to identify the nature of the S2', S3', and S4' binding sites. Alanine at the P2' position was replaced by leucine, phenylalanine, tryptophan, or tyrosine derivs., resulting in Ki values in a significantly lower range compared to I. No upper size limitation or specificity has been found at this position, yet similar replacements at the P3' position, which is occupied naturally by a glycine residue, gave weaker inhibitors.

ACCESSION NUMBER: 1990:36440 CAPLUS
DOCUMENT NUMBER: 112:36440
TITLE: Phosphoramidate peptide inhibitors of human skin fibroblast collagenase
AUTHOR(S): Kortylewicz, Zbigniew P.; Galardy, Richard E.
CORPORATE SOURCE: Dep. Biochem., Univ. Kentucky, Lexington, KY, 40508, USA
SOURCE: Journal of Medicinal Chemistry (1990), 33(1), 263-73
CODEN: JMCMAR; ISSN: 0022-2623
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 112:36440

L12 ANSWER 162 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB Optically pure 3-hydroxyalkanoic acids (I) are prepared by converting I (of 60-85% optical purity) to dibenzylamine salts (II) and recrystg. II. Treatment of (R)-3-hydroxybutanoic acid [prepared from Me (R)-3-hydroxybutanoate (III) of 83% optical purity] with (PhCH2)2NH gave a salt, which was recrystd. from MeCN to give optically pure crystals, which were then converted to optically pure III.

ACCESSION NUMBER: 1990:35296 CAPLUS
DOCUMENT NUMBER: 112:35296
TITLE: Preparation of optically pure 3-hydroxyalkanoic acids as intermediates for drugs and agrochemicals
INVENTOR(S): Kikukawa, Tadashi; Iizuka, Yoshitomi; Tai, Akira
PATENT ASSIGNEE(S): Muraki Buhin Co., Ltd., Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 5 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 01175956	A2	19890712	JP 1988-34	19880104
PRIORITY APPLN. INFO.:			JP 1988-34	19880104

L12 ANSWER 163 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
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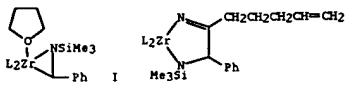

I II

AB A general method for the preparation of zirconocene complexes of imines has been developed. Thus, treatment of PhCH2NH2 with BuLi in Et2O followed by Me3SiCl and more BuLi, and reaction of this solution mixture with Cp2ZrMeCl (Cp = η5-C5H5) in THF afforded 53% (trimethylsilyl)benzaldimine complex I (L = Cp). The x-ray crystal structure of I shows that these complexes should be viewed as metallaaziridines due to significant π-donation from the zirconium center to the π* orbitals of the coordinated imine. These complexes undergo a number of chemo-, regio-, and diastereoselective coupling reactions with unsatd. organic compds. to cleanly form metallacyclic compds., e.g., diazazirconacyclopentene II derived from I (L = Cp) and CH2:CHCH2CH2CH2CN. In situ generation of the complexes followed by coupling with alkynes and hydrolysis affords a general route to geometrically pure allylic amines.

ACCESSION NUMBER: 1989:231793 CAPLUS
DOCUMENT NUMBER: 110:231793
TITLE: Zirconocene complexes of imines. General synthesis, structure, reactivity, and in situ generation to prepare geometrically pure allylic amines
AUTHOR(S): Buchwald, Stephen L.; Watson, Brett T.; Wannamaker, M. Woods; Dewan, John C.
CORPORATE SOURCE: Dep. Chem., Massachusetts Inst. Technol., Cambridge, MA, 02139, USA
SOURCE: Journal of the American Chemical Society (1989), 111(12), 4486-94
CODEN: JACSAT; ISSN: 0002-7863
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 110:231793

L12 ANSWER 164 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB MBF4 (M = NH4, alkali metal) were prepared by the cation exchange reaction of PyH[BF4] (py = pyridine) with MOH or MX (X = halide). The reaction of pyH[BF4] with R3-xNHx (R = alkyl) at room temperature gives rise to R3-xNH1+x[BF4]. The yields are good and the samples are of high purity. The products were characterized by elemental anal., IR and 1H NMR spectroscopy. The spectral data for most of the compds. are reported for the 1st time.

ACCESSION NUMBER: 1989:106911 CAPLUS
DOCUMENT NUMBER: 110:106911
TITLE: A novel synthetic route for the preparation of ammonium and alkali metal tetrafluoroborates and alkyl substituted ammonium tetrafluoroborates using pyridinium tetrafluoroborate as the precursor
AUTHOR(S): Mohamed, K. Syed; Padma, D. K.
CORPORATE SOURCE: Dep. Inorg. Phys. Chem., Indian Inst. Sci., Bangalore, 560 012, India
SOURCE: Indian Journal of Chemistry, Section A: Inorganic, Physical, Theoretical & Analytical (1988), 27A(9), 759-63
CODEN: IJCADU; ISSN: 0376-4710
DOCUMENT TYPE: Journal
LANGUAGE: English

L12 ANSWER 165 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB In the absence of O, the room-temperature photocatalytic conversion of pure primary amines R-NH2 (R-NH2 (R = n-Pr, n-Bu n-pentyl, benzyl) over Pt/TiO2 samples selectively formed sym. N-alkylidene amines. Similarly to other reactions involving H, an optimum Pt content was found. The reaction rate r was proportional to the radiant flux Φ only at relatively low Φ, which indicated that the conversion was monophotonic; at greater Φ, the proportionality of r to Φ1/2 showed that the recombination of the photoproduced charges prevailed. Under these latter conditions, a quantum yield of .apprx.0.015 was calculated (static reactor). In aqueous solns., the same amines led to sym. secondary amines for sufficiently high Pt contents, whereas 1,4-diaminobutane produced pyrrolidine. The variation in the initial rate with the starting concentration was of the Langmuir type with relatively small adsorption consts. for the amines. For aliphatic amines, r decreased with increasing number of C atoms in the presence or absence of H2O. The mechanism is briefly discussed.

ACCESSION NUMBER: 1989:15804 CAPLUS
DOCUMENT NUMBER: 110:15804
TITLE: Photocatalytic formation of symmetrical n-alkylidene amines or secondary amines from primary amines
AUTHOR(S): Tang, F. G.; Courbon, H.; Pichat, P.
CORPORATE SOURCE: Ec. Cent. Lyon, Ecully, 69131, Fr.
SOURCE: Studies in Surface Science and Catalysis (1988), 41(Heterog. Catal. Fine Chem.), 327-36
CODEN: SSCTDM; ISSN: 0167-2991
DOCUMENT TYPE: Journal
LANGUAGE: English

L12 ANSWER 166 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB A simple method is reported for predicting the retention index (RI) of a chemical compound from the number of carbon and carbon equivalent atoms in the mol., the RI increment for atom addition and the group retention factors (GRFs) for substituents and functional groups. Atoms other than carbon such as oxygen, nitrogen, sulfur, chlorine, bromine and iodine are assigned carbon atom equivalency of approx. 1, 1, 2, 2, 3 and 4, resp. and are counted for their contribution towards RI prediction. The GRFs of substituents and functional groups are derived from the RIs of reference compds. and series of homologues. Ring structures, ring fusion, ring connection, iso- and neo-carbons, chain branching and unsatn. are also assigned GRFs. The predicted RIs of a number of alicyclic, aliphatic and aromatic hydrocarbons, primary, secondary and tertiary alcs., phenols, aliphatic amines, aromatic amines, heterocyclics, carboxylic acids, acid esters, aldehydes, ketones, and halogenated compds., are found to be within ±3% of the observed values. The structure-retention index relationship thus developed is extremely useful in the tentative identification of radioactive side products formed in tritium labeling by radiation-induced methods.

ACCESSION NUMBER: 1988:528295 CAPLUS
DOCUMENT NUMBER: 109:128295
TITLE: Prediction of retention indexes. I. Structure-retention index relationship on apolar columns
AUTHOR(S): Peng, C. T.; Ding, S. F.; Hua, R. L.; Yang, Z. C.
CORPORATE SOURCE: Sch. Pharm., Univ. California, San Francisco, CA, 94143, USA
SOURCE: Journal of Chromatography (1988), 436(2), 137-72
CODEN: JOCRAM; ISSN: 0021-9673
DOCUMENT TYPE: Journal
LANGUAGE: English

L12 ANSWER 167 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB RN(CH2OH)CH(CH2CO2-N+H2R2R3)CONHCH(CH2Ph)CO2R1 (R = reductively removable protecting group; R1 = C1-3 alkyl; R2 = H, phenylalkyl; R3 = alkyl, cycloalkyl, phenylalkyl) were prepared as intermediates for Aspartame; they can be purified by recrystn. and stored for a prolonged period of time. Thus, 70.1 N-benzyloxycarbonyl-N-hydroxymethyl-α-aspartylphenylalanine Me ester (I) and 70.1 mmol Me3CNH2 were stirred in EtOAc. The solvent was removed from the mixture and left overnight. The partially crystallized oil was crystallized from EtOAc to give 26.23 g the Me3CNH2 salt of I in 85.5% purity which was recrystd. from MeOH/EtOAc to give the salt with 98.8% purity.

ACCESSION NUMBER: 1988:438248 CAPLUS
DOCUMENT NUMBER: 109:38248
TITLE: Stable crystalline salts of L-N-protected-N-hydroxymethyl-α-aspartyl-L-phenylalanine esters with amines
INVENTOR(S): Tsuda, Makoto; Fujii, Tadashi; Yanagiuchi, Koji; Mitsunobu, Shoichi; Aoki, Shigeru
PATENT ASSIGNEE(S): Nippon Kayaku Co., Ltd., Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 5 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 62283995	A2	19871209	JP 1986-125898	19860602

PRIORITY APPLN. INFO.: JP 1986-125898 19860602
OTHER SOURCE(S): CASREACT 109:38248

L12 ANSWER 168 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

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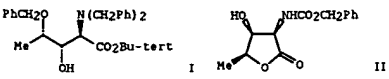

AB Enantiomerically pure tert-Bu 2-amino-2,5-dideoxy-L-lyxo-pentanoate (I) was synthesized via the highly diastereoselective MgBr2 mediated addition of silylketene acetal (PhCH2)2NCH:C(OSiMe3)(OBu-tert) to (S)-O-benzyllactic aldehyde. The synthesis of γ-lactone II, a known intermediate in the synthesis of L-daunosamine and L-vancosamine, is also described.

ACCESSION NUMBER: 1988:132213 CAPLUS
DOCUMENT NUMBER: 108:132213
TITLE: Stereoselective synthesis of tert-butyl 2-amino-2,5-dideoxy-L-lyxo-pentanoate: formal synthesis of L-daunosamine
AUTHOR(S): Banfi, Luca; Cardani, Silvia; Potenza, Donatella; Scolastico, Carlo
CORPORATE SOURCE: Ist. Chim. Org., Univ. Genova, Genoa, 16132, Italy
SOURCE: Tetrahedron (1987), 43(10), 2317-22
CODEN: TETRAB; ISSN: 0040-4020
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 108:132213

L12 ANSWER 169 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

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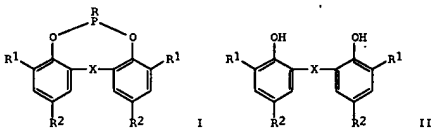

AB Thirteen title compds. I [R = Cl, NHPh, NPh2, N(CH2Ph)2, piperidino, piperazino; R1 = Me3C, H, Cl, 2-methylcyclohexyl; R2 = Me, Me3C, Cl; X = CH2, CHCCl3, CHC6H4Cl-o, S] were prepared in 76-88% yields by cyclizing phenols II with PCl3 followed optionally by treatment with amines. I are intermediates for preparing polymer stabilizers.

ACCESSION NUMBER: 1987:576111 CAPLUS
DOCUMENT NUMBER: 107:176111
TITLE: Synthesis of the acid chlorides of eight-membered cyclic phosphorous acids and their derivatives
AUTHOR(S): Mukmeneva, N. A.; Kadyrova, V. Kh.; Zharkova, V. M.; Cherkasova, D. A.; Voskresenskaya, O. V.
CORPORATE SOURCE: Kazan. Khim.-Tekhnol. Inst., Kazan, USSR
SOURCE: Zhurnal Obshchei Khimii (1986), 56(10), 2267-71
CODEN: ZOKHA4; ISSN: 0044-460X
DOCUMENT TYPE: Journal
LANGUAGE: Russian
OTHER SOURCE(S): CASREACT 107:176111

L12 ANSWER 170 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB The title stabilization was made by milling or dispersion of pigments with an equimolar mixture of C6-24 fatty acid(s) and C1-10 amine(s) including morpholine in nonaq. solvent of surface tension >25 dynes/cm. Thus, 350 parts leafing-type Al paste was mixed with 125 parts solution from palmitic acid 25.6, 2-ethylbutylamine 10.1, and xylene 220.3 parts to give a dispersion which (30 parts) was mixed with 270 parts Acrydic 45-468-Super Beckamine J820 mixture, thinned with xylene to Ford Cup Number 4 viscosity 16 s at 20°, and stored in a sealed can, showing leafing stability (DIN 55923) 2 mo.

ACCESSION NUMBER: 1987:479581 CAPLUS
DOCUMENT NUMBER: 107:79581
TITLE: Metal pigment leafing stabilization
INVENTOR(S): Ishijima, Shizuo; Hayashi, Yukio
PATENT ASSIGNEE(S): Asahi Chemical Industry Co., Ltd., Japan
SOURCE: Jpn. Tokkyo Koho, 4 pp.
CODEN: JAXXAD
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 62024460	B4	19870528	JP 1977-125090	19771020
JP 63234072	A2	19880929	JP 1988-13596	19880126

PRIORITY APPLN. INFO.: JP 1977-125090 19771020

L12 ANSWER 171 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB Cr-carbene complexes containing the (:C(H)NR2) group were prepared by reaction
of Vilsmeier's salts with Cr(CO)52-. These carbenes were remarkably air stable and resistant to attack by nucleophiles. Photoreaction of these complexes with imines, oxazines, oxazolines, imidates, thiazines, and thiazolines produced β-lactams in fair to good yield. In most cases trans stereochem. was observed Representative dibenzylamino-β-lactams were debenzylated to produce β-lactams having a free NH2 group α to the lactam carbonyl group.

ACCESSION NUMBER: 1987:101443 CAPLUS
DOCUMENT NUMBER: 106:101443
TITLE: Synthesis of amino-β-lactams by the photolytic reaction of imines with pentacarbonyl[(dibenzylamino)carbene]chromium(0)
AUTHOR(S): Borel, Christian; Hegedus, Louis S.; Krebs, Jurg; Satoh, Yoshitaka
CORPORATE SOURCE: Dep. Chem., Colorado State Univ., Fort Collins, CO, 80523, USA
SOURCE: Journal of the American Chemical Society (1987), 109(4), 1101-5
CODEN: JACSAT; ISSN: 0002-7863
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 106:101443

L12 ANSWER 172 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB 1H and 11B NMR spectroscopy was applied to mono- and bisborane adducts derived from aryl-, benzyl-, phenethyl- and phenylenediamines, but no simple relation was established between the spectroscopic data and the nature of the N-B bond. Comparative studies of the affinity of aromatic amines to BH3 by equilibrium reactions may be of great value in establishing a
scale of relative basicity.

ACCESSION NUMBER: 1987:94878 CAPLUS
DOCUMENT NUMBER: 106:94878
TITLE: Studies on aromatic amine boranes by boron-11 and proton NMR
AUTHOR(S): Camacho, C.; Paz-Sandoval, M. A.; Contreras, R.
CORPORATE SOURCE: Cent. Invest. Estud. Avanzados, IPN, Mexico City, Mex.
SOURCE: Polyhedron (1986), 5(11), 1723-32
CODEN: PLYHDE; ISSN: 0277-5387
DOCUMENT TYPE: Journal
LANGUAGE: English

L12 ANSWER 173 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
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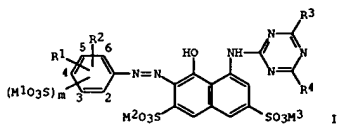

I

AB The azo dyes I (M1-M3 = H, alkali metal, NH4, quaternary ammonium; m = 1, 2; R1, R2 = C1-3 alkyl, C1-3 alkoxy, halogen, H; R3, R4 = C6-18 amine, alkoxyalkylamine, alkanolamine) are useful in nonclogging aqueous jet-printing
inks. H acid was condensed with cyanuric chloride, and this intermediate was coupled with diazotized orthanilic acid and then condensed with (2-ethylhexyloxy)propylamine and (PhCH2)2NH. A jet-printing ink containing this dye 3.5, polyethylene glycol 8, glycerol 1, Bu(OCH2CH2)2OH 1, N-methylpyrrolidone 24, (HOCH2CH2)3N 2, and H2O 50.5 had good storage stability at 0°, at 50° had jetting
stability 90 days, good image clarity, and gave no bleeding from printings on wood-free paper in water.

ACCESSION NUMBER: 1986:628503 CAPLUS
DOCUMENT NUMBER: 105:228503
TITLE: Azo dyes for aqueous jet-printing inks
INVENTOR(S): Kawashita, Hideo; Ota, Mitsuhiro
PATENT ASSIGNEE(S): Taoka Chemical Co., Ltd., Japan; Sumitomo Chemical Co., Ltd.
SOURCE: Eur. Pat. Appl., 24 pp.
CODEN: EPXXDW
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.		DATE
EP 194885	A1	19860917	EP 1986-301823		19860313
EP 194885	B1	19890607			
R: BE, CH, DE, FR, GB, IT, LI, NL					
JP 62156168	A2	19870711	JP 1986-53443		19860311
US 4771129	A	19880913	US 1986-839153		19860313
PRIORITY APPLN. INFO.:			JP 1985-51408	A	19850314
			JP 1985-200382	A	19850909

OTHER SOURCE(S): CASREACT 105:228503

L12 ANSWER 174 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
GI For diagram(s), see printed CA Issue.
AB A series of new ligands and the corresponding technetium-99m chelates based on diamide dimercaptide donor groups I (X = CH2CH2, C6H4, CH2CHMe, CH2COCH2, etc.) were synthesized as derivs. of technetium-99m 1,2-bis(2-thioacetamideo)ethane, a complex shown to be excreted by renal tubular secretion. Chelation with 99mTc resulted in single radiochem. products or the expected number of stereoisomers. They were purified by high performance liquid chromatog. and evaluated in mice as potential renal tubular function agents. The in vivo properties were sensitive to the presence of functional groups, the positional isomerism of the carboxylate group functionality, and the chelate ring stereochem. of the ligand. The presence of Me groups slowed renal transit and decreased renal specificity. Cyclohexyl rings fused to the ethylene bridge of the center chelate ring decreased renal excretion while aromatic rings essentially abolished renal excretion. Slow hepatobiliary clearance was observed as an alternate mode of excretion. Polar groups, increased renal excretion rates and specificity in a stereochem. dependent manner. 99mTc chelates of 1,3-bis(2-thioacetamido)-2-hydroxypropane, 3,4-bis(2-thioacetamideo)butanoate and 1,8-dimercapto-2,7-dioxo-3,6-diazanonanoate were identified as promising new renal radiopharmaceuticals.

ACCESSION NUMBER: 1986:625972 CAPLUS
DOCUMENT NUMBER: 105:225972
TITLE: Tissue distribution properties of technetium-99m-diamide-dimercaptide complexes and potential use as renal radiopharmaceuticals
AUTHOR(S): Kasina, Sudhakar; Fritzberg, Alan R.; Johnson, Dennis L.; Eshima, Dennis
CORPORATE SOURCE: Sch. Med., Univ. Utah, Salt Lake City, UT, 84132, USA
SOURCE: Journal of Medicinal Chemistry (1986), 29(10), 1933-40
CODEN: JMCMAR; ISSN: 0022-2623
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 105:225972

L12 ANSWER 175 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB Airborne isophorone diisocyanate (I) [4098-71-9] is determined by drawing air
through solns. of 1-(o-methoxyphenyl)piperazine [35386-24-4], N-(p-nitrobenzyl)propylamine [103796-64-1], and dibenzylamine [103-49-1], forming stable derivs. suitable for reverse-phase high-performance liquid chromatog. with UV detection. In-situ derivatization of I during sampling stabilized the samples. The structures of the derivs. formed by reaction with the secondary amines were authenticated by IR, NMR, and elemental anal. These derivs. were purified, and their use for calibration purposes is proposed in preference to calibration with the extremely unstable I.

ACCESSION NUMBER: 1986:538892 CAPLUS
DOCUMENT NUMBER: 105:138892
TITLE: High performance liquid chromatographic analysis of airborne isophorone diisocyanate and the authentication of analytical standards
AUTHOR(S): Wu, Weh S.; Huang, Lolita K.; Gaind, Virindar S.
CORPORATE SOURCE: Occup. Health Lab., Ontario Minist. Labour, Weston, ON, M9P 3T1, Can.
SOURCE: American Industrial Hygiene Association Journal (1958-1999) (1986), 47(8), 482-7
CODEN: AIHAAP; ISSN: 0002-8894
DOCUMENT TYPE: Journal
LANGUAGE: English

L12 ANSWER 176 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
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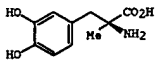

I

AB In order to characterize the in vivo metabolic fate of the antihypertensive agent α-methyldopa (I) [555-30-6] the urine of α-methyldopa-treted rats was examined with the aid of a direct insertion probe chemical ionization mass spectral assay. The mass spectrum of the sample obtained by chromatog. purification followed by treatment with ethanolic hydrochloric acid and pentafluoropropionic anhydride displayed an intense ion at m/z 812, consistent with the β-ethoxy-N,O,O,O-tetrakispentafluoropropionyl derivative of 6-hydroxy-α-methylnorepinephrine, a potential aromatic hydroxylation product of the known α-methyldopa metabolite α-methylnorepinephrine. Comparison of this spectrum with the spectrum obtained with the corresponding synthetic 6-hydroxy-α-methylnorepinephrine [104024-06-8], however, ruled out this possibility. A more thorough examination of the mass spectral data established that the ion
at m/z 812 observed with the metabolic species was due to the formation of an
unexpected adduct ion between a known metabolite of α-methyldopa and an impurity ion formed from a common constituent of urine. This paper summarizes the characterization of this adduct ion.

ACCESSION NUMBER: 1986:507876 CAPLUS
DOCUMENT NUMBER: 105:107876
TITLE: Unexpected adduct ion formation under chemical ionization conditions
AUTHOR(S): Musson, Donald G.; Halldin, Magnus M.; Karashima, Deiji; Castagnoli, Neal, Jr.
CORPORATE SOURCE: Sch. Pharm., Univ. California, San Francisco, CA, 94143, USA
SOURCE: Biomedical & Environmental Mass Spectrometry (1986), 13(6), 287-91
CODEN: BEMSEN; ISSN: 0887-6134
DOCUMENT TYPE: Journal
LANGUAGE: English

L12 ANSWER 177 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB In the removal of N compds., O compds., and olefins from such synthetic-petroleum fractions as naphtha (represented by PhMe [108-88-3]) on zeolite 13X, both N and O compds. are strongly adsorbed, but such low-basicity compds. as 2,4,6-collidine [108-75-8] are poorly adsorbed from PhMe even in the absence of any competition. Olefins are able to compete with N compds. in adsorption only at very high concns.

ACCESSION NUMBER: 1986:500117 CAPLUS
DOCUMENT NUMBER: 105:100117
TITLE: The competitive adsorption of fuel-type compounds on zeolite 13X
AUTHOR(S): Jean, G.; Chantal, P.; Ahmed, S.; Sawatzky, H.
CORPORATE SOURCE: Energy Res. Lab., Ottawa, ON, K1A 0G1, Can.
SOURCE: Preprints of Papers - American Chemical Society, Division of Fuel Chemistry (1986), 31(3), 262-5
CODEN: ACFPAI; ISSN: 0569-3772
DOCUMENT TYPE: Journal
LANGUAGE: English

L12 ANSWER 178 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB A method for the determination of 1,3-bis(isocyanatomethyl)-cyclohexane (H6XDI)
[38661-72-2] in air is based on H6XDI collection using a midget impinger, conversion into a stable urea derivative with dibenzylamine, and anal. by high performance liquid chromatog. with UV detection at 254 nm. The collection efficiency is ≥98% and the detection limit is 0.16 μg H6XDI, which corresponds to 1.0 ppb in a 20 L air sample.

ACCESSION NUMBER: 1986:94334 CAPLUS
DOCUMENT NUMBER: 104:94334
TITLE: Determination of 1,3-bis(isocyanatomethyl)cyclohexane(H6XDI) in working atmosphere by high performance liquid chromatography
AUTHOR(S): Matsuura, Yoshikatsu
CORPORATE SOURCE: Chem. Prod. Div., Takeda Chem. Ind. Ltd., Osaka, 532, Japan
SOURCE: Takeda Kenkyushoho (1985), 44(1/2), 124-30
CODEN: TAKHAA; ISSN: 0371-5167
DOCUMENT TYPE: Journal
LANGUAGE: English

L12 ANSWER 179 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB R4COCHR3CH2CONR1R2 [R1, R2 = H, C1-20 alkyl, cycloalkyl, C7-20 aralkyl, C6-14 aryl, each (un)substituted with C1-6 alkoxy or alkoxy, F, Cl, Br,iodo, or with C1-6 alkyl or alkoxy substituted with F, Cl, Br, or iodo; R3 = (un)substituted C1-20 alkyl; R4 = OR5, NR5R6; R5, R6 = R1 or R2], useful as antioxidants, stabilizing agents for polymers, and as synthons for insecticides, acaricides, herbicides, fungicides, and for pharmacol. and physiol. active compds. (no data), were prepared by treating R7CONR1R2 (R7 = C3-30 alkenyl) with HX (X = OR5, NR5R6) and with CO in the presence of Co compds. and optionally ≥1 tertiary N bases at elevated temps. and pressures. A mixture of MeCH:CHCONEt2 (I), PhOH, pyridine, and Co2(CO)8 was treated with CO containing 2% H2 in a shaking autoclave at 170°/150 bar 45 min to give 91.5% conversion of I with 56.9% yield C5 dicarboxylic acid derivs., of which 87.7% was PhO2CCHMeCH2CONEt2 and 12.3% was PhO2C(CH2)3CONEt2.
ACCESSION NUMBER: 1986:68580 CAPLUS
DOCUMENT NUMBER: 104:68580
TITLE: Substituted succinic acid amides
INVENTOR(S): Kadelka, Juergen; Schwarz, Hans Helmut
PATENT ASSIGNEE(S): Bayer A.-G. , Fed. Rep. Ger.
SOURCE: Eur. Pat. Appl., 33 pp.
CODEN: EPXXDW
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 2
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 143303	A2	19850605	EP 1984-112433	19841016
EP 143303	A3	19860430		
R: CH, DE, FR, GB, IT, LI				
DE 3339386	A1	19850530	DE 1983-3339386	19831029
DE 3420112	A1	19851205	DE 1984-3420112	19840530
PRIORITY APPLN. INFO.:			DE 1983-3339386 A	19831029
			DE 1984-3420112 A	19840530
OTHER SOURCE(S):	CASREACT 104:68580			

L12 ANSWER 180 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB The title salts, dissolving rapidly in hydrocarbons to give concentrated, stable solns., are prepared by heating NH4 molybdates with carboxylic acids in the presence of amines with distillation of H2O. Thus, stirring NH4 molybdate 5.5, naphthenic acid 18.5, and Bu3N 4.0 parts at 200° for 10 h with distillation of H2O gave a salt dissolving in 20 mL PhEt to give a solution containing 6% Mo, which formed no precipitate during 1 mo in air. Stirring this salt 5, C3H6 46, and a 35% PhEt solution of PhCH(Me)OOH (I) 50 parts at 120° for 1 h gave propylene oxide with selectivity 86.5% (based on I) and I conversion 99.6%; compared with 86.8 and 95.9, resp., when com. Mo naphthenate was used.
ACCESSION NUMBER: 1986:51236 CAPLUS
DOCUMENT NUMBER: 104:51236
TITLE: Hydrocarbon-soluble salts of molybdenum for epoxidation of olefins
INVENTOR(S): Usui, Masahiro; Higashio, Yasuhiko
PATENT ASSIGNEE(S): Atlantic Richfield Co., USA
SOURCE: Eur. Pat. Appl., 18 pp.
CODEN: EPXXDW
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 155156	A2	19850918	EP 1985-301628	19850308
EP 155156	A3	19861008		
EP 155156	B1	19881130		
R: BE, DE, FR, GB, IT, NL				
JP 60191020	A2	19850928	JP 1984-46145	19840309
JP 05085485	B4	19931207		
US 4593012	A	19860603	US 1985-708480	19850305
ES 541092	A1	19861216	ES 1985-541092	19850308
ES 550962	A1	19870216	ES 1986-550962	19860116
US 5017712	A	19910521	US 1988-217119	19880708
PRIORITY APPLN. INFO.:			JP 1984-46145 A	19840309
			US 1985-708480 A3	19850305
			US 1986-816037 B1	19860103

L12 ANSWER 181 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB The title compds., useful as antioxidants and polymer stabilizing agents, were prepared by reaction of RCOX1 [R = α,β- or β,γ-unsatd. unbranched or branched, (un)substituted C3-30 alkyl; X1 = NH2, NHR1, NR1R2; R1, R2 = C1-20 alkyl or cycloalkyl, C7-20 aralkyl, or C6-14 aryl each (un)substituted with C1-6 alkyl and(or) alkoxy and(or) F, Cl, Br, and(or) iodo] with CO and HX2(X2 = OR3, NH2, NHR3, NR3R4; R3, R4 = R1) in the presence of Co compds. and optionally in the presence of ≥1 tertiary N bases at elevated temps. and pressures. A mixture of N,N-diethylcrotonamide (I), PhOH, pyridine, and Co2(CO)8 was treated with CO containing 2% H at 170°/150 bar 45 min to give 91.5% conversion of I and 56.9% yield of C5 dicarboxylic acid derivs., of which 87.7% was PhO2CCHMeCH2CONEt2 and 12.3% PhO2C(CH2)3CONEt2.
ACCESSION NUMBER: 1986:19410 CAPLUS
DOCUMENT NUMBER: 104:19410
TITLE: Diverse derived 2-substituted succinic acids
INVENTOR(S): Kadelka, Juergen; Schwarz, Hans Helmut
PATENT ASSIGNEE(S): Bayer A.-G. , Fed. Rep. Ger.
SOURCE: Ger. Offen., 30 pp.
CODEN: GWXXBX
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 2
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 3339386	A1	19850530	DE 1983-3339386	19831029
EP 143303	A2	19850605	EP 1984-112433	19841016
EP 143303	A3	19860430		
R: CH, DE, FR, GB, IT, LI				
JP 60112747	A2	19850619	JP 1984-223111	19841025
US 4588833	A	19860513	US 1984-665226	19841026
PRIORITY APPLN. INFO.:			DE 1983-3339386 A	19831029
			DE 1984-3420112 A	19840530

L12 ANSWER 182 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB The use of model compds. combined with gas chromatog. characterizes complex adsorption systems, to yield information on the adsorption mechanism. The possibility of using adsorbents for the selective removal of N compds. from petroleum fractions is demonstrated. The adsorbent is ilmenite treated with bromide. Coker kerosine is purified. The extent of removal is high for basic N compds. but low for acidic/neutral N compds.
ACCESSION NUMBER: 1985:580626 CAPLUS
DOCUMENT NUMBER: 103:180626
TITLE: Separation of nitrogenous-type compounds from synthetic crudes
AUTHOR(S): Jean, G.; Poirier, M.; Sawatzky, H.
CORPORATE SOURCE: Hydrocarbon Process. Res. Lab., CANMET, Ottawa, ON, K1A 0G1, Can.
SOURCE: Separation Science and Technology (1985), 20(7-8), 541-53
CODEN: SSTEDS; ISSN: 0149-6395
DOCUMENT TYPE: Journal
LANGUAGE: English

L12 ANSWER 183 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB Hpy[PF6] (py = pyridine) reacts at room temperature with RNH2, R2NH, and R3N (R = alkyl), forming RNH3[PF6], R2NH2[PF6], and R3NH[PF6], reesp., while with R4NX it gives R4N[PF6]. The yields are good and the samples are of high purity. The compds. were characterized by elemental analyses, IR and 1H NMR spectroscopy. The spectral data of most of the compds. are reported for the 1st time.

ACCESSION NUMBER: 1985:533826 CAPLUS
DOCUMENT NUMBER: 103:133826
TITLE: Preparation of alkyl substituted ammonium hexafluorophosphates using pyridinium hexafluorophosphate
AUTHOR(S): Mohamed, K. Syed; Padma, D. K.; Kalbandkeri, R. G.; Murthy, A. R. Vasudeva
CORPORATE SOURCE: Dep. Inorg. Phys. Chem., Indian Inst. Sci., Bangalore, 560 012, India
SOURCE: Indian Journal of Chemistry, Section A: Inorganic, Physical, Theoretical & Analytical (1985), 24A(3), 195-8
CODEN: IJCADU; ISSN: 0376-4710
DOCUMENT TYPE: Journal
LANGUAGE: English

L12 ANSWER 184 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB Cationic, lipid-soluble organic compds. may interfere with cation-mediated membrane transport processes. Thus, small intestinal absorption may be influenced by lipophilic organic cations. Therefore, a series of arylalkylamines was studied in the concentration range from 0.5 to 20 mM for their effect on the transport of various monosaccharides and leucine in the rat small intestine in vitro by means of the tissue accumulation technique. Whereas the monophenyl substituted monoamines (e.g. benzylamine, 2-phenylethylamine, and 3-phenylpropylamine) did not show a significant effect on the active transport, the corresponding α,α-di-Ph derivs. exhibited a strong inhibition of the active transport of the sugars and the amino acid. These monoamines and drugs of similar structure (e.g. benzoctamine and diphenhydramine) exhibited a mixed or noncompetitive type of inhibition which correlated quite well with their octanol-water partition coeffs. In contrast, di- or triamines (e.g. harmaline, imipramine, and pyrilamine) revealed a rather pure competitive type of inhibition. These findings tentatively suggest a different mode of action on the active transport by lipid-soluble organic amines according to the mol. charge distribution. In addition, membrane vesicles were used to examine the effect of the different amines on the sucrase activity. Regarding the cation-dependent hydrolysis of sucrose, however, no distinct pattern developed.

ACCESSION NUMBER: 1985:451988 CAPLUS
DOCUMENT NUMBER: 103:51988
TITLE: In vitro inhibition of rat small intestinal absorption by lipophilic organic cations
AUTHOR(S): Elsenhans, Bernd; Blume, Roland; Lembcke, Bernhard; Caspary, Wolfgang F.
CORPORATE SOURCE: Inst. Pharmakol. Toxikol., Univ. Muenchen, Munich, D-8000/2, Fed. Rep. Ger.
SOURCE: Biochimica et Biophysica Acta (1985), 813(1), 25-32
CODEN: BBACAQ; ISSN: 0006-3002
DOCUMENT TYPE: Journal
LANGUAGE: English

L12 ANSWER 185 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB IR, UV, and NMR of the title primary and secondary amino title compds. show that they do not exist as imines or nitronic acids but do contain an intramol. H bond which stabilizes the (Z)-configuration, solubility studies show that these H-bonded enamines are highly polar due to a large resonance contribution from the delocalized imonium ion. This resonance interaction is enhanced in the case of the tertiary amino title compds.

ACCESSION NUMBER: 1984:610453 CAPLUS
DOCUMENT NUMBER: 101:210453
TITLE: Structural study of α-amino-β-nitrostilbenes
AUTHOR(S): Allade, Irenee; Dubois, Pierre; Levillain, Pierre; Viel, Claude
CORPORATE SOURCE: Lab. Pharm. Chim. II, Fac. Pharm., Chatenay-Malabry, Fr.
SOURCE: Bulletin de la Societe Chimique de France (1983), (11-12, Pt. 2), 339-44
CODEN: BSCFAS; ISSN: 0037-8968
DOCUMENT TYPE: Journal
LANGUAGE: French
OTHER SOURCE(S): CASREACT 101:210453

L12 ANSWER 186 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB Oxidation of primary and secondary amines with (RC6H4SO2O)2 [R = 4-NO2, 3-CF3 (I)] were examined Optimal results were obtained with I as the oxidant and KOH as the promoting base in AcOEt at -78°. Under these conditions, yields of carbonyl products were generally higher than other methods for both amine types. The stability of the intermediate imine is of great importance in determining the success of the conversion.

ACCESSION NUMBER: 1984:570217 CAPLUS
DOCUMENT NUMBER: 101:170217
TITLE: The oxidation of amines with sulfonyl peroxide. 8. Oxidative deamination of amines by arylsulfonyl peroxides
AUTHOR(S): Hoffman, Robert V.; Kumar, Anil
CORPORATE SOURCE: Dep. Chem., New Mexico State Univ., Las Cruces, NM, 88003, USA
SOURCE: Journal of Organic Chemistry (1984), 49(21), 4011-14
CODEN: JOCEAH; ISSN: 0022-3263
DOCUMENT TYPE: Journal
LANGUAGE: English
OTHER SOURCE(S): CASREACT 101:170217

L12 ANSWER 187 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB The adsorption was studied of model N compds. on natural sulfides and brominated ilmenite . N compds. are adsorbed preferentially on acidic centers of these minerals; a general correlation between the basicity of the N compds. and the extent of their adsorption was observed The brominated ilmenite, which has bromides of Ti and Fe (Lewis acids) on the surface, is a much better adsorbent than the untreated ilmenite or natural sulfides, such as pyrrhotite.
ACCESSION NUMBER: 1984:554442 CAPLUS
DOCUMENT NUMBER: 101:154442
TITLE: Removal of synthetic crude nitrogenous compounds using waste minerals
AUTHOR(S): Jean, G.; Poirier, M.; Sawatzky, H.
CORPORATE SOURCE: Energy Res. Lab., CANMET, Ottawa, ON, K1A 0G1, Can.
SOURCE: Preprints of Papers - American Chemical Society, Division of Fuel Chemistry (1984), 29(6), 243-8
CODEN: ACFPAI; ISSN: 0569-3772
DOCUMENT TYPE: Journal
LANGUAGE: English

L12 ANSWER 188 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB Equilibration between 2',5'- and 3',5'-di-O-benzoyladenosine derivs. on Wakogel C-300 and Merck 60 silica gel gave mixts. predominantly containing the latter. Adsorbed water and hydroxyl functions of silicic acid were important for the equilibration through the acyl migration from the 2'- and 3'-position. The effect of substituents at the N6-position of adenosine on the equilibration was also investigated.
ACCESSION NUMBER: 1983:72652 CAPLUS
DOCUMENT NUMBER: 98:72652
TITLE: Partial protection of carbohydrate derivatives. Part 9. Equilibration between 2',5'- and 3',5'-di-O-benzoyladenosine derivatives substituted at the N6-position, on silica gel
AUTHOR(S): Sakairi, Nobuo; Rahman, Dalilur; Tanaki, Kazuaki; Ishido, Yoshiharu
CORPORATE SOURCE: Fac. Sci., Tokyo Inst. Technol., Tokyo, 152, Japan
SOURCE: Nucleosides & Nucleotides (1982), 1(2), 99-110
CODEN: NUNUD5; ISSN: 0732-8311
DOCUMENT TYPE: Journal
LANGUAGE: English

L12 ANSWER 189 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB Among the major products of electron-beam radiolysis of alkylarom. amines (N,N-dinonylaniline; N,N-dibenzyldodecylamine; N-benzyldinonylamine) in octane solns. were secondary amines formed by dissociation of C-N bond and tertiary amines formed by substitution of H, alkyl or aryl at α-C atom (with respect to N) in the parent amine mol. by a solvent radical. O strongly increased the efficiency of the product formation and introduction of octanol (30 weight%) decreased the efficiency of the tertiary amine formation. In solns. containing HNO3 the efficiency of the secondary amine formation sharply increased and the tertiary amine formation was fully quenched.
ACCESSION NUMBER: 1983:63224 CAPLUS
DOCUMENT NUMBER: 98:63224
TITLE: Stable products of the radiolysis of solutions of tertiary alkylaromatic amines and their nitrate salts
AUTHOR(S): Kersulis, V.; Egorov, G. F.; Zagorets, P. A.
CORPORATE SOURCE: Inst. Elektrokhim., Moscow, USSR
SOURCE: Khimiya Vysokikh Energii (1982), 16(6), 505-10
CODEN: KHVKAO; ISSN: 0023-1193
DOCUMENT TYPE: Journal
LANGUAGE: Russian

L12 ANSWER 190 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB Conversion of aliphatic primary and secondary amines into metal dithiocarbamate chelates was examined for high-performance liquid chromatog. determination of these amines. Characteristic chromatograms based on the difference in the rate of ligand exchange were obtained for different central metal ions. When Hg(II) chelates were tested, trace determination of individual secondary amines was possible because only the peaks of binary complexes corresponding to each amine appeared. When Ni(II) and Pd(II) chelates were tested, peaks appeared for ternary complexes as well as for binary complexes. This phenomenon was applied to determining optical purity of antiasthmatic ephedrine isomers in Chinese crude drugs.
ACCESSION NUMBER: 1982:79161 CAPLUS
DOCUMENT NUMBER: 96:79161
TITLE: High-performance liquid chromatographic determination of organic substances by metal chelate derivatization. I. Dithiocarbamate chelates of aliphatic amines
AUTHOR(S): Moriyasu, Masataka; Hashimoto, Yohei; Endo, Masaru
CORPORATE SOURCE: Kobe Women's Coll. Pharm., Kobe, 658, Japan
SOURCE: Bulletin of the Chemical Society of Japan (1981), 54(11), 3369-73
CODEN: BCSJA8; ISSN: 0009-2673
DOCUMENT TYPE: Journal
LANGUAGE: English

L12 ANSWER 191 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB H2NCR2P(O)(OH)2 (R = H or Me) and HN[CH2P(O)(OH)2]2 were obtained by catalytic hydrogenation of the [benzyl(amino)alkyl]phosphonic acids. The reduction occurred with quant. yields and pure acids were easily isolated.

ACCESSION NUMBER: 1980:446775 CAPLUS
DOCUMENT NUMBER: 93:46775
TITLE: New preparative method for aminomethylphosphonic, aminoisopropylphosphonic and iminobis(methylenephosphonic) acids
AUTHOR(S): Szczepaniak, W.; Kuczynski, K.
CORPORATE SOURCE: Inst. Chem., Univ. A. Mickiewicz, Poznan, 780, Pol.
SOURCE: Phosphorus and Sulfur and the Related Elements (1979), 7(3), 333-7
CODEN: PREEDF; ISSN: 0308-664X
DOCUMENT TYPE: Journal
LANGUAGE: French
OTHER SOURCE(S): CASREACT 93:46775

L12 ANSWER 192 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

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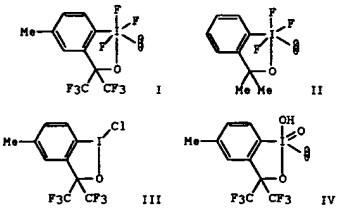

I II III IV

AB Stable alkoxyaryltrifluoroperiodinanes I and II were prepared by oxidation of the resp. parent iodo alcs. 5,2-MeIC6H3C(CF3)2OH and 2-IC6H4CMe2OH with excess CF3OF. The stability and low reactivity of I and II are ascribed to the strong stabilizing influence of the 5-membered ring. The reaction of I with Me3SiCl gives the corresponding iodine(III) species, III, and chlorine. I is hydrolyzed with aqueous base to give a species thought to be iodinane oxide (IV). I is a selective reagent for the oxidation of primary and secondary amines or alcs. bearing α hydrogens to the corresponding aldehyde or ketone. In contrast to iodine pentafluoride, I does not further oxidize the product aldehydes to acids. tert-Butylamine is oxidized by I to give 1,1,1',1'-tetramethylazoethane. PhMgBr reacts with I to give PhF. Possible mechanisms for these selective oxidns. are discussed. It is suggested that the stabilizing structural features of I make it a tamed analog of IF5.

ACCESSION NUMBER: 1980:22441 CAPLUS
DOCUMENT NUMBER: 92:22441
TITLE: Synthesis and reactions of stable alkoxyaryltrifluoroperiodinanes. A "tamed" analog of iodine pentafluoride for use in oxidations of amines, alcohols, and other species
AUTHOR(S): Amey, Ronald L.; Martin, J. C.
CORPORATE SOURCE: Sch. Chem. Sci., Univ. Illinois, Urbana, IL, 61801, USA
SOURCE: Journal of the American Chemical Society (1979), 101(18), 5294-9
CODEN: JACSAT; ISSN: 0002-7863
DOCUMENT TYPE: Journal
LANGUAGE: English

L12 ANSWER 193 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB RR1NCOSR2 (R, R1 = alkyl, alkoxy, alkenyl, cycloalkyl, hydroxyalkyl, Ph, CH2Ph; NRR1 = heterocyclic; R2 = alkyl, optionally substituted CH2Ph) were prepared by treating COS with RR1NH2 and treating RR1NCOSH.HNRR1 with R2X (X = halogen). Thus, HNEt2 was treated COS to give 59.9% Et2NCOSH.HNEt2 which was treated with 4-ClC6H4CH2Cl to give 99.5% Et2NCOSCH2C6H4Cl-4, 98.95% pure.

ACCESSION NUMBER: 1979:507825 CAPLUS
DOCUMENT NUMBER: 91:107825
TITLE: Thiol carbamates
INVENTOR(S): Sato, Zenichi; Tabuchi, Fumiya; Takagi, Kaiichiro; Imamiya, Yoji
PATENT ASSIGNEE(S): Ihara Chemical Industry Co., Ltd., Japan
SOURCE: Ger. Offen., 24 pp.
CODEN: GWXXBX
DOCUMENT TYPE: Patent
LANGUAGE: German
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 2844305	A1	19790517	DE 1978-2844305	19781011
DE 2844305	C2	19880121		
JP 54073732	A2	19790613	JP 1977-137424	19771116
JP 61002656	B4	19860127		
US 4248779	A	19810203	US 1978-948346	19781004
IN 149403	A	19811128	IN 1978-CA1128	19781018
AU 7841003	A1	19800501	AU 1978-41003	19781024
AU 521869	B2	19820506		
CA 1103265	A1	19810616	CA 1978-315330	19781031
BR 7807443	A	19790724	BR 1978-7443	19781110
IL 55915	A1	19820331	IL 1978-55915	19781110
ES 475077	A1	19790501	ES 1978-475077	19781114
DD 139713	C	19800116	DD 1978-209079	19781114
HU 175382	P	19800728	HU 1978-IA833	19781114
CS 203936	P	19810331	CS 1978-7420	19781114
PL 114064	B1	19810131	PL 1978-210932	19781115
RO 76088	P	19810228	RO 1978-95684	19781115
SU 1041032	A3	19830907	SU 1978-2688147	19781116

PRIORITY APPLN. INFO.: JP 1977-137424 A 19771116

L12 ANSWER 194 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB The effects of the 3 N substituents on the reactivities of aliphatic amines were analyzed by free energy-related substituent consts. and regression anal. In bonding with CHCl3 and in charge-transfer complexation with I2, electronic and steric effects of the 3 N substituents were quant. separated by the equation log K = ρ*Σσ* + a1Esc(R1) + a2Esc(R2) + a3Esc(R3) + c, where K is the equilibrium constant, ρ*, a1, a2 and a3 are susceptibility consts., and c is the intercept. The Σσ* is the sum of the Taft σ* values of the 3 N substituents. Esc(R1), Esc(R2) and Esc(R3) are, resp., the Hancock corrected steric consts. of N substituents R1, R2 and R3, where Esc(R1) ≥ Esc(R2) ≥ Esc(R3). Examination of literature data suggest a general applicability of the present procedure to various reactivities of aliphatic amines.

ACCESSION NUMBER: 1979:490949 CAPLUS
DOCUMENT NUMBER: 91:90949
TITLE: Quantitative separation of electronic and steric substituent effects in reactions between aliphatic amines and electron acceptors
AUTHOR(S): Takayama, Chiyozo; Fujita, Toshio; Nakajima, Minoru
CORPORATE SOURCE: Dep. Agric. Chem., Kyoto Univ., Kyoto, 606, Japan
SOURCE: Journal of Organic Chemistry (1979), 44(16), 2871-9
CODEN: JOCEAH; ISSN: 0022-3263
DOCUMENT TYPE: Journal
LANGUAGE: English

L12 ANSWER 195 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB The synergic extraction of Co2+ from aqueous perchlorate by thenoyltrifluoroacetone (I) and 8 amines, e.g. tri-n-octylamine, in CHCl3 was examined The extracted product was shown to be a 1:2:1 Co-I-amine complex. Co-amine bonding was confirmed by IR and UV spectra. The stability sequence of aryl complexes is dibenzylamine > benzylamine ≤ tribenzylamine. For long-chain alkyl tertiary amines the log of the adduct formation consts. increase linearly with increasing Taft inductive constant

ACCESSION NUMBER: 1978:536468 CAPLUS
DOCUMENT NUMBER: 89:136468
TITLE: Synergic extraction of cobalt(II) by thenoyltrifluoroacetone and some amine extractants in chloroform
AUTHOR(S): Aly, H. F.; Raieh, M.; Mohamed, S.; Abdel-Rassoul, A. A.
CORPORATE SOURCE: Nucl. Chem. Dep., At. Energy Establ., Cairo, Egypt
SOURCE: Journal of Inorganic and Nuclear Chemistry (1978), 40(3), 567-70
CODEN: JINCAO; ISSN: 0022-1902
DOCUMENT TYPE: Journal
LANGUAGE: English

L12 ANSWER 196 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

AB The extraction of Fe2+, Co2+, Cu2+, and Zn2+ from aqueous perchlorate of ionic strength 0.1 ((H, Na)ClO4) into a mixture of thenoyltrifluoroacetone (HTTA) and dibenzylamine (DBA) in chloroform was studied. The extraction of the different cations increases by more than 10³ in the presence of DBA. Slope anal. of the extraction results assumed a general formula of M(TTA)2·DBA for the extractable adduct. A stability order of Fe(TTA)2·DBA > Co(TTA)2·DBA > Zn(TTA)2·DBA > Cu(TTA)2·DBA was established.

ACCESSION NUMBER: 1978:28455 CAPLUS
DOCUMENT NUMBER: 88:28455
TITLE: Synergic extraction of divalent iron, cobalt, copper and zinc with thenolytrifluoroacetone-dibenzylamine in chloroform
AUTHOR(S): Aly, H. F.; Raieh, M.; Mohamed, S.; Abdel-Rassoul, A. A.
CORPORATE SOURCE: Nucl. Chem. Dep., At. Energy Establ., Cairo, Egypt
SOURCE: Journal of Radioanalytical Chemistry (1977), 41(1), 65-71
CODEN: JRACBN; ISSN: 0022-4081
DOCUMENT TYPE: Journal
LANGUAGE: English

L12 ANSWER 197 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

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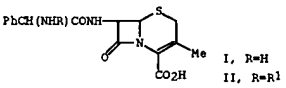

I, R=H
II, R=R1

AB Cephalexin (I) was prepared by treatment of crude (II R1 = NH2-protecting groups) with (PhCH2)2NH, separation and purification of the formed (PhCH2)2NH salts, liberation of the free acids II, and removal of the protecting groups. Thus, a mixture of 3.86 g Li D-α-tert-butoxycarbonylaminophenylacetate and SO3/DMF was stirred 20 min, added to 2.14 g 7-amino-3-methyl-3-cephem-4-carboxylic acid in H2O (pH 7.5 with NaHCO3) at 5-10°, and the whole stirred 30 min to give 5.6 g crude 7β-(D-α-tert-butoxycarbonylamino-α-phenylacetamido)-3-methyl-3-cephem-4-carboxylic acid (III). To III in AcOEt-Et2O was added 84 ml (PhCH2)2NH to precipitate 5.85 g III.(PhCH2)2NH salt. III.(PhCH2)2NH (2 g) in aqueous AcOEt was made pH 3.0 with citric acid to give III. III in CH2Cl2 was stirred with 5 ml concentrated HCl 1 hr at room temperature to give 2.1 g I.

ACCESSION NUMBER: 1977:5474 CAPLUS
DOCUMENT NUMBER: 86:5474
TITLE: Cephalosporin derivative
INVENTOR(S): Sugimoto, .Shingo; Nakabayashi, Satoru; Katano, Kiyoaki; Fukatsu, Shunzo; Seki, Shigeo
PATENT ASSIGNEE(S): Meiji Confectionary Co., Ltd., Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 4 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 51059889	A2	19760525	JP 1974-131132	19741115
JP 60046117	B4	19851014		
PRIORITY APPLN. INFO.:			JP 1974-131132 A	19741115

L12 ANSWER 198 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

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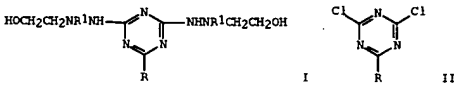

AB Triazines I [R = NR2R3, SR2 (R2 = C6-18 saturated or unsatd. alkyl, cyclohexyl, CH2C6H5-nR4n, C6H5-nR4n, n = 0-5, R4 = halo, MeO, EtO, HO, cyano, Me, Bu, etc.; R3 = H, R2; R1 = H, CH2CH2OH] were prepared by treating II with hydroxyethylhydrazines H2NNR1CH2CH2OH III. I are antioxidants for polyamides or polyurethanes and prevents discoloration of basic dyes. Thus, 27.6 parts II (R = dibenzylamino), prepared from cyanuric chloride and (PhCH2)2NH, was treated with 36.5 parts III (R1 = H) in aqueous dioxane at 20-30° and heated at 50-80° to give I (R = dibenzylamino, R1 = H). This (3%) was added to cellulose diacetate and the film dyed with Kaylon Fast Blue FN. On exposure to NOx, it underwent no discoloration. Among 6 more I prepared were (R, R1 given): (PhCH2)2N, CH2CH2OH; dilaurylamino, H; distearylamino, CH2CH2OH; stearylthio, H.

ACCESSION NUMBER: 1976:592774 CAPLUS
DOCUMENT NUMBER: 85:192774
TITLE: 2-Substituted 4,6-bis(hydroxyethylhydrazino)-s-triazines
INVENTOR(S): Moriga, Hiroyuki
PATENT ASSIGNEE(S): Teijin, Ltd., Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 6 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 51054575	A2	19760513	JP 1974-127810	19741106
JP 56022865	B4	19810527		
PRIORITY APPLN. INFO.:			JP 1974-127810 A	19741106

L12 ANSWER 199 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
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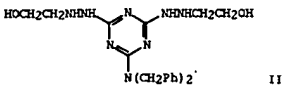

II

AB Urethane rubbers resistant to yellowing by N oxides, Cl bleach, and light contained bis[2-(2-hydroxyethyl)hydrazine]-s-triazine derivs. For example, polytetramethylene glycol was polymerized with diphenylmethane diisocyanate, and the prepolymer in DMF was treated with aqueous N2H4 and Et2NH and then 2% TiO2 to give 30% rubber solution (I). Cyanuric chloride [108-77-0] was condensed with dibenzylamine [103-49-1] to give 2-dibenzylamino-4,6-dichloro-s-triazine [47301-29-1] which was treated with (2-hydroxyethyl)hydrazine [109-84-2] to give 2,4-bis[2-(2-hydroxyethyl)hydrazino]-6-(dibenzylamino)-s-triazine (II) [60188-59-2]. The I solution was mixed with 3 phr II, cast, gelled with water, dried at 100° for 30 min, and heat-treated at 120° for 20 min to give yellowing-resistant film.

ACCESSION NUMBER: 1976:495542 CAPLUS
DOCUMENT NUMBER: 85:95542
TITLE: Yellowing-resistant urethane rubber compositions
INVENTOR(S): Moriga, Hiroyuki
PATENT ASSIGNEE(S): Teijin, Ltd., Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 9 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.		DATE
JP 51053552	A2	19760512	JP 1974-127809		19741106
PRIORITY APPLN. INFO.:			JP 1974-127809	A	19741106

L12 ANSWER 200 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB Racemic norepinephrine was synthesized with three D atoms on the alkyl chain. The deuteration was accomplished by D/H exchange on the intermediate, 2-(dibenzylamino)-3',4'-dihydroxyacetophenone, followed by reduction of the keto moiety and cleavage of the benzyl-protecting groups with D gas. Noradrenalone was also shown to be a possible intermediate for the incorporation of 18O into norepinephrine.

ACCESSION NUMBER: 1976:58827 CAPLUS
DOCUMENT NUMBER: 84:58827
TITLE: Synthesis of stable isotope labeled norepinephrine
AUTHOR(S): Murphy, R. C.
CORPORATE SOURCE: Med. Sch., Univ. Colorado, Denver, CO, USA
SOURCE: Journal of Labelled Compounds (1975), 11(3), 341-7
CODEN: JLCAAI; ISSN: 0022-2135
DOCUMENT TYPE: Journal
LANGUAGE: English

L12 ANSWER 201 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB Polyester nonwoven fabric-based urethane rubber leather substitutes with improved durability and yellowing resistance contained 0.1-5% 2-(cyclohexylamino)-4,6-dimorpholino-s-triazine (II) [51304-98-4] and/or 2-(dibenzylamino)-4,6-bis(2,2-dimethylhydrazino)-s-triazine [51304-96-2]. The rubbers were prepared from 4,4'-diphenylmethane diisocyanate, polyethylene glycol, and poly(hexamethylene neopentyl adipate)diol or poly(neopentyl tetramethylene adipate)diol [neopentyl glycol content in total diol ≤40%; polyesterdiol mol. weight 500-4000; polyester/polyethylene glycol >4]. Cyanuric chloride [108-77-0] was condensed with morpholine [110-91-8] and then cyclohexanamine [108-91-8] to give I; II was obtained by condensation of cyanuric chloride with dibenzylamine [103-49-1] and then 1,1-dimethylhydrazine [57-14-7].

ACCESSION NUMBER: 1976:45578 CAPLUS
DOCUMENT NUMBER: 84:45578
TITLE: Urethane rubber leather substitutes with improved durability and yellowing resistance
INVENTOR(S): Mimura, Masahisa; Ohkawa, Nobuo
PATENT ASSIGNEE(S): Teijin Kodore Co., Ltd., Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 7 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.		DATE
JP 50125001	A2	19751001	JP 1974-30562		19740319
JP 56044193	B4	19811017			
PRIORITY APPLN. INFO.:			JP 1974-30562	A	19740319

L12 ANSWER 202 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB Hexachlorocyclotriphosphazatriene, N3P3Cl6, with (PhCH2)2NH gave N3P3Cl6-n[N(CH2Ph)2]n (n = 1, 2) and with PhCH2NH2 it gave N3P3Cl6-n(NHCH2Ph)n [n = 1, 2 (2 isomers), 4, 6]. Mixed (dimethylamino)(dibenzylamino) and -(benzylamino) derivs. were prepared and assigned structures by NMR spectroscopy. The role of steric effects in the reactions of N3P3Cl6 with bulky nucleophiles was discussed. The stability of N3P3Cl[N(CH2Ph)2](NMe2)4 arose from protection of the P-Cl bond from nucleophilic attack by the bulky geminal N(CH2Ph)2 substituent.

ACCESSION NUMBER: 1976:38151 CAPLUS
DOCUMENT NUMBER: 84:38151
TITLE: Phosphorus-nitrogen compounds. XLI. Reactions of hexachlorocyclotriphosphazatriene with dibenzylamine and benzylamine. Importance of steric effects. Isolation of a stable chloro(dibenzylamino)tetrakis(dimethylamino) derivative
AUTHOR(S): Masood-ul-Hasan; Shaw, Robert A.; Woods, Michael
CORPORATE SOURCE: Dep. Chem., Birkbeck Coll., London, UK
SOURCE: Journal of the Chemical Society, Dalton Transactions: Inorganic Chemistry (1972-1999) (1975), (21), 2202-7
CODEN: JCDTBI; ISSN: 0300-9246
DOCUMENT TYPE: Journal
LANGUAGE: English

L12 ANSWER 203 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
GI For diagram(s), see printed CA Issue.
AB A series of 41 title compds., prepared by alkylation of the appropriate secondary amine, were tested in vitro as inhibitors of fibrinoligase [9067-75-8]. Some of the compds. were among the most active fibrinoligase inhibitors known, with 5-[bis(4-chlorobenzyl)amino]pentylamine fumarate (1 fumarate) [55097-48-8] being twice as active as monodansylcadaverine [10121-91-2]. The dibenzylamino moiety at one end of the mol. and primary amino group at the other end the compound could function both as a pseudo donor substrate and noncompetitive alkylating inhibitor. Structure-activity relations are discussed.

ACCESSION NUMBER: 1975:588192 CAPLUS
DOCUMENT NUMBER: 83:188192
TITLE: Fibrin-stabilizing factor inhibitors. 12. 5-Dibenzylaminopentylamine and related compounds, a new type of FSF [fibrin-stabilizing factor] inhibitors
AUTHOR(S): Hoffmann, Kurt Juergen; Stenberg, Pal; Ljunggren, Christine; Svensson, Uno; Nilsson, J. Lars G.; Eriksson, Olle; Hartkoorn, Ann; Lunden, Ragnar
CORPORATE SOURCE: Fac. Pharm., Univ. Uppsala, Uppsala, Swed.
SOURCE: Journal of Medicinal Chemistry (1975), 18(3), 278-84
CODEN: JMCMAR; ISSN: 0022-2623
DOCUMENT TYPE: Journal
LANGUAGE: English

L12 ANSWER 204 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB The improved stability of the magnetic recording materials was achieved by including an organic corrosion inhibitor in the composition The material consists of a nonmagnetizable support covered with a magnetizable layer made up of metal particles (Fe, Ni or Co or alloys of these, each particle of which may be covered with a layer of Cr) dispersed in a nonmagnetizable binding material. To this magnetizable layer is added at least 0.0001 g. equivalent of a nonsterically hindered aliphatic amine. The amine must have a pKa of at least 8, measured in aqueous solution at 25°. Tertiary amines, polyurethanes and tris-2,4,6-(dimethylaminomethyl)phenol are particularly favored. A surface active acid may also be added to disperse the particles. For example, acicular 300 Å particles of Fe (75), Co (5-8), coated with Cr (3-4%) were mixed with tridecylpolyethylene oxide phosphoric ester and PhMe. Tris(dimethylaminomethyl)phenol (2%) was added, along with a polymeric binding material (30%). Films of the material of 30 μ thick were withdrawn by scraping. These were dried in air and heated at 66°. After a corrosion test at 66° and 80% humidity for 18 hr no signs of corrosion were seen, while a similar sample which did not contain tris(dimethylaminomethyl)phenol showed considerable corrosion over all its surface.

ACCESSION NUMBER: 1975:541118 CAPLUS
DOCUMENT NUMBER: 83:141118
TITLE: Magnetic recording composition based on fine metallic particles, with improved stability towards the environment
INVENTOR(S): Heikkinen, Duane G.; Kanten, Thomas M.
PATENT ASSIGNEE(S): Minnesota Mining and Manufacturing Co.
SOURCE: Fr. Demande, 17 pp.
CODEN: FRXXBL
DOCUMENT TYPE: Patent
LANGUAGE: French
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
FR 2212597	A1	19740726	FR 1973-46783	19731228
CA 1003707	A1	19770118	CA 1973-187875	19731211
NL 7317577	A	19740704	NL 1973-17577	19731221
JP 49099004	A2	19740919	JP 1974-4397	19731228
AU 7364016	A1	19750703	AU 1973-64016	19731228
DE 2365292	A1	19740718	DE 1973-2365292	19731231
IT 1002574	A	19760520	IT 1973-54673	19731231
GB 1459750	A	19761231	GB 1973-60194	19731231
US 4074012	A	19780214	US 1975-608916	19750829
PRIORITY APPLN. INFO.:			US 1973-320630 A	19730102

L12 ANSWER 205 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB PhP(O)Cl2 with (PhCH2)2NH (LH) in organic solvents at room temperature gave PhP(O)L(OEt), PhP(O)(OEt)2, PhP(O)ClL, and [PhP(O)L]2O. PhP(O)ClL was not isolated but with RNH2 (R = Et, PhCH2) gave PhP(O)L(NHR). PhP(S)Cl2 with LH gave PhP(S)ClL, PhP(S)L(OEt), PhP(S)L(NHCH2Ph), and 2 isomers of [PhP(S)L]2O. PhP(S)Cl2 with LH in wet C6H6 gave LH2+ [PhPSLO]-. The EtO compds. only formed in stabilized CHCl3. PMR showed that many CH2 groups were intrinsically asym.

ACCESSION NUMBER: 1974:505640 CAPLUS
DOCUMENT NUMBER: 81:105640
TITLE: Phosphorus-nitrogen compounds. XXXVIII. Reactions of phenylphosphonic dichloride and phenylphosphonothioic dichloride with dibenzylamine
AUTHOR(S): Healy, James D.; Shaw, Robert A.; Smith, Barry C.; Thakur, Chandramauleshwar P.; Woods, Michael
CORPORATE SOURCE: Dep. Chem., Birkbeck Coll., London, UK
SOURCE: Journal of the Chemical Society, Dalton Transactions: Inorganic Chemistry (1972-1999) (1974), (12), 1286-90
CODEN: JCDTBI; ISSN: 0300-9246
DOCUMENT TYPE: Journal
LANGUAGE: English

L12 ANSWER 206 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB A 2 step synthesis of dl-PhCH(OH)CH(NH2)14CH3 (I) from PhCOCH2N(CH2Ph)2 and 14CH3I is described. After purification by chromatog. on an ion exchange resin column AG 50W-X2 I.HCl is obtained with a radioactive overall yield of 31% based on Ba14CO3, sp. activity: 55 mCi/mmole. The anal. by paper electrophoresis in conjunction with the paper and thin-layer chromatog. enables control of radiochem. purity of I.

ACCESSION NUMBER: 1974:477595 CAPLUS
DOCUMENT NUMBER: 81:77595
TITLE: Synthesis of methyl-carbon-14 labeled dl-norephedrine
AUTHOR(S): Nguyen Hoang Nam; Lucas, P. ; Pichat, Louis
CORPORATE SOURCE: Serv. Mol. Marquees, CEN Sacly, Gif-sur-Yvette, Fr.
SOURCE: Journal of Labelled Compounds (1974), 10(1), 49-57
CODEN: JLCAAI; ISSN: 0022-2135
DOCUMENT TYPE: Journal
LANGUAGE: French

L12 ANSWER 207 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB Addition of 0.001-0.3 weight% benzylamine [100-46-9]-Cu halide complex or dibenzylamine [103-49-1]-cupric chloride complex (I) to nylon 6 melt or a mixture containing hexamethylenediammonium adipate improved the thermal stability and resistance to uv degradation of nylon fiber without causing coloration of the fiber, which was useful for tire cords and belts. Thus, nylon 6 [25038-54-4] containing 0.05 weight% benzylamine-cupric chloride complex(2:1) [14434-96-9] (prepared from 17g cupric chloride [7447-39-4] and 18.8g benzylamine was mixed 15 min at 290.deg. without discoloration. The tensile strength retention for a fiber prepared by melt spinning a mixture containing nylon 6 and 0.06 wt% I was 94% after heating 4 hr at 180.deg., compared to 28% for a fiber prepared without I. Benzylamine-cuprous iodide [7681-65-4] complex, benzylamine-cupric bromide [7789-45-9] complex, and benzylamine-cuprous chloride [7758-89-6] complex were also used.

ACCESSION NUMBER: 1974:414583 CAPLUS
DOCUMENT NUMBER: 81:14583
TITLE: Stabilized nylon composition
INVENTOR(S): Fujii, Shigeru; Saito, Isoo
PATENT ASSIGNEE(S): Toray Industries, Inc.
SOURCE: Jpn. Tokkyo Koho, 4 pp.
CODEN: JAXXAD
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 48020017	B4	19730618	JP 1969-44520	19690607
PRIORITY APPLN. INFO.:			JP 1969-44520	19690607

L12 ANSWER 208 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB The powder static susceptibilities of the crystalline stable free radical 1,1-diphenyl-2-picrylhydrazyl and of samples recrystd. from various solvents were measured at room temperature The value of the static susceptibility was also computed from microchem. anal. data and from ESR data. The samples recrystd. from different solvents show different values of susceptibility. This is interpreted on the basis of the exchange interaction and lone pair properties of the solvents.

ACCESSION NUMBER: 1973:471613 CAPLUS
DOCUMENT NUMBER: 79:71613
TITLE: Static magnetic susceptibility of 1,1-diphenyl-2-picryl hydrazyl recrystallized powders
AUTHOR(S): Misra, B. N.; Gupta, S. K.
CORPORATE SOURCE: Dep. Phys., Allahabad Univ., Allahabad, India
SOURCE: Revue de Physique Appliquee (1973), 8(2), 117-19
CODEN: RPHAAN; ISSN: 0035-1687
DOCUMENT TYPE: Journal
LANGUAGE: English

L12 ANSWER 209 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB The purpose of the additives is to extend the range of c.ds. with which good deposits can be obtained. A suggested additive mixture consists of 0.1-2 weight } Ph2NH with a PhOH-glucose condensate making up the remainder (up to 5 weight }) of the bath. The bath itself consists of SnSO4 55, C6H4(OH)SO3H 30, and H2O 915 parts. The range of c.ds. is 5-50 A/dm2 and bath temperature is 50°. A highly synergistic effect is obtained.

ACCESSION NUMBER: 1972:521546 CAPLUS
DOCUMENT NUMBER: 77:121546
TITLE: Additives for tin electroplating baths
PATENT ASSIGNEE(S): Ciba-Geigy A.-G.
SOURCE: Fr. Demande, 15 pp.
CODEN: FRXXBL
DOCUMENT TYPE: Patent
LANGUAGE: French
FAMILY ACC. NUM. COUNT: 2
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
FR 2095375	A5	19720211	FR 1971-22461	19710621
GB 1339133	A	19731128	GB 1970-29819	19710528
PRIORITY APPLN. INFO.:			GB 1970-29819 A	19700619

L12 ANSWER 210 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB Poly(ethylene sulfide) is stabilized against thermal degradation by addition of KSeCN, KSCN, and (or) NH4SCN, an amine, and a metal oxide. Thus, a mixture of 2835 ml tetrahydrofuran, 9.838 ml H2O and Et2Zn (H2O-Et2Zn molar ratio 1:1) was stirred under N, added under N to a mixture of 2946 g ethylene sulfide (I) in 23.56 kg petroleum ether at 25.5 ± 1.1 °, stirred 1 hr, centrifuged, the resulting polymer dried in vacuo at 71-82°, and powdered to give 590 g polymeric catalyst (II). II was added under N to a mixture of 27.285 kg I in 77.24 kg petroleum ether, the mixture heated during 1.5 hr to 80 ± 2.75°, kept 2 hr at this temperature, cooled to 38°, centrifuged, and the separated polymer dried for 4 hr in vacuo at 80° to give 80% poly(ethylene sulfide) (III) with tensile strength 615 kg/cm2, elongation 3.48%, and modulus 27.3 + 10-3 kg/cm2, which changed to 133 kg/cm2, 0.45%, and 28.56% after 10 days' aging in air at 121°. III containing 1.5% KSeCN when molded gave a product with initial tensile strength 640 kg/cm2, elongation 5.36%, and modulus 23.52 + 10-3 kg/cm2, as compared to 649 kg/cm2, 3.66%, and 29.4 + 10-3 kg/cm2 after 10 days aging at 121°. III containing KSeCN 1, dibenzylethylenediamine 3, phenyl-β-naphthylamine 1, and ZnO 0.2% was molded to give a product with initial tensile strength 684 kg/cm2, elongation 24.36%, and modulus 17.29 + 10-3 kg/cm2, which changed to 651 kg/cm2, 7.45%, and 17.99 + 10-3 kg/cm2 after agin g 10 days in air at 121°. Other amines used were dibenzylamine, pentaethylenehexamine, and (4-H2NCH2CH2-NHCH2C6H4)2O. TiO2, MgO or CaO may be used instead of ZnO.

ACCESSION NUMBER: 1970:112278 CAPLUS
DOCUMENT NUMBER: 72:112278
TITLE: Stabilized poly(ethylene sulfide)
INVENTOR(S): Ellerstein, Stuart M.
PATENT ASSIGNEE(S): Thiokol Chemical Corp.
SOURCE: Fr., 26 pp.
CODEN: FRXXAK
DOCUMENT TYPE: Patent
LANGUAGE: French
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
FR 1576906		19690801		
DE 1769918			DE	
GB 1222705			GB	
US 3519596		19700000	US	
PRIORITY APPLN. INFO.:			US	19670810

L12 ANSWER 211 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB N.M.R. spectra (60 MHz.) were recorded on 0.1-1% solns. of 66 amine compds. (15 primary, 18 secondary, 10 tertiary, 23 aromatic) in CHCl3 at 32°. The location of the CHCl3-band vs. Me3Si was determined, and the stability consts. of CHCl3-amine complexes calculated Results are tabulated. For all nonaromatic amines, the chemical shift of the CHCl3-complex was dependent on the basicity, or the sum of the polar consts. of the substituents on the N. For all the aromatic amines, in addition to the complexation with N, an association with π electrons of the aromatic ring is involved, and becomes increasingly more significant with increasing steric hindrance or decreasing basicity of the amine group.
ACCESSION NUMBER: 1968:414620 CAPLUS
DOCUMENT NUMBER: 69:14620
TITLE: Nuclear magnetic resonance studies on the hydrogen bond. II. Chemical shift of chloroform-amine complexes
AUTHOR(S): Suhr, Harald
CORPORATE SOURCE: Univ. Tuebingen, Tuebingen, Fed. Rep. Ger.
SOURCE: Journal of Molecular Structure (1968), 1(4/5), 295-303
CODEN: JMOSB4; ISSN: 0022-2860
DOCUMENT TYPE: Journal
LANGUAGE: German

L12 ANSWER 212 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB Manufacture of cladding Zircaloys implies starting with 98% ore and silica-free
zirconia before dehafnization and metallurgical elaboration. Dehafnization of fed zirconia still containing 1.4% HfO2 was studied. The usual organophosphorus and amine solvents were exand, in view of enhancing maximum loading charge and introducing cheaper com. varieties. Bu3PO4 as a 60% solution is suggested after examining numerous diluents (odorless kerosine, iso-BuCOMe, xylol, n-hexane, benzene, cyclohexane, toluene) besides white spirit. Examined variables were the time of contacting (1-5 min.) and the concns. of free HNO3 (5 to 8 molar), fed zirconium (5-100 g./l., and salting-out agents (about 3.5 molar nitrates). Longchain aliphatic and aromatic amines examined include: Armeen C, S, T, TD, and HTD, and FB-Amine 10, 12, 16, 17, and 18. Tri- and dibenzylamine, triaurylamine hydrochlorides, and sulfate liquors were studied, and the effect of lowering temperature, increasing acidity, and changing diluents were examined
ACCESSION NUMBER: 1966:426818 CAPLUS
DOCUMENT NUMBER: 65:26818
ORIGINAL REFERENCE NO.: 65:4951g-h
TITLE: Nuclear-grade zirconium from Egyptian zircon placers
AUTHOR(S): Farah, M. Y.; El-Yamani, I. S.
CORPORATE SOURCE: U.A.R. At. Energy Estab., Inshas
SOURCE: Proc. Intern. Conf. Peaceful Uses At. Energy, 3rd, Geneva, 1964 (1965), Volume 9, 131-8
From: Nucl. Sci. Abstr. 18(21), 4992(1964).
DOCUMENT TYPE: Report
LANGUAGE: English

L12 ANSWER 213 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
GI For diagram(s), see printed CA Issue.
AB Polymers of acrolein have found limited use because they are readily oxidized in air, resulting in mol.-weight degradation. Organic amines with vapor pressures of <1 mm. at 30° and having the formula XX1N(X4NX2)nX3 are used to stabilize acrolein polymers, especially polyacrolein. X, X1, X2, and X3 are H, C1-18 alkyl groups, or C6-18 aryl groups. X4 may be a divalent C1-10 alkylene group or a divalent C6-10 arylene group; n is an integer (0-5). The amines used may be primary, secondary, or tertiary. Heterocyclic secondary amines of the formula I may also be used, where z is 0 or 1; Y is a CH2 group, a secondary amine, S, or O; and Ar is an arylene group. For example, 5 g. of polyacrolein powder was stirred with 20 ml. of an acetone solution containing 0.01 g. phenyl-2-napthylamine as I. After evaporation of the acetone, the mixture containing
0.2 weight % stabilizer was placed in an oven at 140°F. Reduced viscosities were measured at 30° by using a solution of 0.2 g. of stabilized polymer in 100 ml. of a saturated solution of SO2 in H2O. A polyacrolein sample containing no stabilizer had an initial reduced viscosity of 4.0. After 1, 2, and 3 weeks, resp., the reduced viscosities were 1.3, 0.8, and 0.5. The sample stabilized with I had an initial reduced viscosity of 4.0 and a reduced viscosity of 2.4 after 3 weeks.
ACCESSION NUMBER: 1966:44680 CAPLUS
DOCUMENT NUMBER: 64:44680
ORIGINAL REFERENCE NO.: 64:8408c-f
TITLE: Stabilization of acrolein polymers with secondary amines
INVENTOR(S): Welch, Frank J.
PATENT ASSIGNEE(S): Union Carbide Corp.
SOURCE: 3 pp.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 3225000		19651221	US	19610609

L12 ANSWER 214 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB A differential vapor pressure technique was used to study the self-association
of certain acids and bases in several nonhydrogen bonding solvents. In 1,2-dichloroethane, the self-association of benzoic acid is markedly decreased
by ortho substitution with bromine and hydroxy and methoxy groups. Ortho substitution in phenol with nitro and methoxy groups has the same effect, which is attributed in part to stabilization of the monomeric form by intramol. H bonding. Acetamide appears to form a relatively stable trimer, but amines undergo little association in 1,2-dichloroethane. Benzoic acid shows significant association in nitromethane, but none in acetonitrile which has virtually the same dielec. constant The lack of association in acetonitrile is attributed to H bonding between acid and solvent, stabilizing the monomer.
ACCESSION NUMBER: 1965:450092 CAPLUS
DOCUMENT NUMBER: 63:50092
ORIGINAL REFERENCE NO.: 63:9110a-b
TITLE: A differential vapor pressure study of the self-association of acids and bases in 1,2-dichloroethane and certain other solvents
AUTHOR(S): Coetzee, J. F.; Lok, Rose Mei-Shun
CORPORATE SOURCE: Univ. of Pittsburgh, Pittsburgh, PA
SOURCE: Journal of Physical Chemistry (1965), 69(8), 2690-6
CODEN: JPCHAX; ISSN: 0022-3654
DOCUMENT TYPE: Journal
LANGUAGE: English

L12 ANSWER 215 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB The title compound NaB(p-ClC6H4)4 (I), was synthesized and purified . Aqueous I may be used to identify qual. alkali ions and some basic N compds. (as the HCl salts). Two ml. of an aqueous 1% solution of I as the mixed Na-Mg salt gave a heavy precipitate with each of the following, at 0.05M concentration: K+, NH4+, Rb+, Cs+, 1-phenylethylamine, EtNH2, Et2NH, (PhCH2)2NH, atropine (II), (CH2)6N4, 1,6-H2N(CH2)6NH2, glycine, Bu4NCl, benzidine (III), BuNH2 (IV), and brucine (V) (each base as its HCl salt). III-V, and quinine, form stoichiometric compds. with I. Ba++, Cu++, Ni++, Ca++, Cd++, and Co++ gave no ppts. with the mixed Na-Mg salt; C5H5N gave a light precipitate; PhNH2 and II formed ppts. that were unsuitable as derivs. K+, 5 γ/ml. and 100 γ/ml., is detected by forming a trace of precipitate with 2 ml. of 1% NaBPh4 (VI), or with I, resp. The solubility of KB(p-Cl-C6H4)4 in H2O at 25° and pH 3.7, 6.7, or 6.8 is 6.0, 6.5, and 7.5 + 10-4M, resp. Because of this relatively high solubility of the K salt, recoveries were low.
ACCESSION NUMBER: 1965:413472 CAPLUS
DOCUMENT NUMBER: 63:13472
ORIGINAL REFERENCE NO.: 63:2392b-d
TITLE: Tetraaryl borates. I. The preparation and reagent properties of sodium tetrakis(p-chlorophenyl)borate
AUTHOR(S): Cassaretto, Frank P.; McLafferty, John J.; Moore, Carl E.
CORPORATE SOURCE: Loyola Univ., Chicago
SOURCE: Analytica Chimica Acta (1965), 32(4), 376-80
CODEN: ACACAM; ISSN: 0003-2670
DOCUMENT TYPE: Journal
LANGUAGE: English

L12 ANSWER 216 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB cf. CA 53, 3530f. Exposure of apples to 1 mole BuOAc in 5000 moles air gave no increase in scale, but produced a marked taint. Cyclohexane, cyclohexene, C6H6, and d-limonene applied as vapors, and C16H34 and C14H29CH:CH2 applied to the surface in EtOH, reduced scald at appropriate concns. The last 3 at high concns. produced scald-like injury. During storage in oiled wraps, cuticle oil and ursolic acid were transferred to the wraps, and mineral oil to the apples. A more volatile minor fraction of the mineral oil contributed to scald control. Ph2NH controlled scald better than PhCH2NHPh, (PhCH2)2NH, or dicyclohexylamine (in decreasing order of effectiveness) when used as dips in EtOH. PH2NH reduced volatile ester production at 1°, increased it at 20°, increased the production of less volatile esters of the lipid coating, and stabilized a pigment in the lipid coating. Quercitin applied in EtOH solution reduced scald, but cyanidin did not.
ACCESSION NUMBER: 1964:443633 CAPLUS
DOCUMENT NUMBER: 61:43633
ORIGINAL REFERENCE NO.: 61:7604a-b
TITLE: Superficial scald, a functional disorder of stored apples. II. Promoters and inhibitors
AUTHOR(S): Huelin, F. E.
CORPORATE SOURCE: Commonwealth Sci. Ind. Res. Org., North Ryde, Australia
SOURCE: Journal of the Science of Food and Agriculture (1964), 15(4), 227-36
CODEN: JSFAAE; ISSN: 0022-5142
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L12 ANSWER 217 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
GI For diagram(s), see printed CA Issue.
AB cf. CA 56, 5862b. I and II were prepared by MnO2 oxidation of the appropriate dihydrazone. HgO oxidation of the dihydrazone of p-C6H4(CHO)2 gave III. Structural differences influence the stability of these compds. III reacted with AcOH to give p-C6H4(CH2OAc)2. Treatment of III with Ph3P gave p-C6H4(CH:NN:PPh3)2. The crystals of all the bisdiazo compds. were strongly dichroic.
ACCESSION NUMBER: 1964:417920 CAPLUS
DOCUMENT NUMBER: 61:17920
ORIGINAL REFERENCE NO.: 61:2996d-e
TITLE: Dicarbenes. Some isolable bisdiazoalkanes
AUTHOR(S): Murray, Robert W.; Trozzolo, Anthony M.
CORPORATE SOURCE: Bell Telephone Labs., Inc., Murray Hill, NJ
SOURCE: Journal of Organic Chemistry (1964), 29(5), 1268-70
CODEN: JOCEAH; ISSN: 0022-3263
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L12 ANSWER 218 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
AB Reaction mixts., from contacting aminoethylpiperazine with SiO2-Al2O3, are distilled to give a fraction (b. 160-90°), the fraction is cooled to 10-40°, a portion of the distillate fraction crystallized to give the title compound, the mother liquor concentrated, and the concentrate, which is rich in triethylenediamine (I), recycled to the distn. zone in an apparatus which is described. Thus, a fraction, b. 160-90°, containing 60-75% I is placed in a kettle and heated at 70°, the mixt cooled to .apprx.25°, and the slurry that forms centrifuged to give 484 g. 99.0 weight-% I and 682 g. mother liquor containing 37.3 weight-% I.
ACCESSION NUMBER: 1964:52324 CAPLUS
DOCUMENT NUMBER: 60:52324
ORIGINAL REFERENCE NO.: 60:9148a-c
TITLE: Purification of triethylenediamine
INVENTOR(S): Muhlbauer, Herbert G.; Cour, Thomas H.
PATENT ASSIGNEE(S): Jefferson Chemical Co., Inc.
SOURCE: 4 pp.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 3120525		19640204	US	19610518

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AB Reaction of R2NH with HCHO can led to R2NCH2OH (I) and (R2N)2CH2 (II). The extent of the existence of the intermediate I in the reaction of R2NH with HCHO was investigated calorimetrically. HCHO (1 mole) was added to 2 moles R2NH and the temperature rise, ΔT1, measured in a simple, Nernst-type calorimeter. A 2nd mole of HCHO was added and the rise in temperature, ΔT2, measured. Data corrected for heats of dilution of amine in H2O were tabulated for reactions at 0 and 30°. The ΔT1 and ΔT2 values were readily explained by considering the equilibrium involved in the reactions R2NH + HCHO .rdblhar. R2NCH2OH; R2NCH2OH + R2NH .rdblhar. (R2N)2CH2 + H2O. The data indicated that equilibrium favored II at both temps. and that generally the ratio ΔT1/ΔT2 was greater at 30° than at 5°, indicating the greater stability of II over that of I. EtNHCH2CH2OH and (HOCH2CH2)2NH had low ΔT1/ΔT2 ratios (0.33:0.17 and 0.56:0.16, and 0.81:0.31 and 0.45:0.23 at 0 and 30°, resp.) owing to formation of the corresponding oxazolidines, 3-ethyloxazolidine, b. 122°, n22D 1.4322, and 3-(β-hydroxyethyl)oxazolidine, b4.7 93°, n30D 1.4753. The low values for ΔT1 (0.28 and 0.05 at 0 and 30°) for (PhCH2)2NH made it impossible to decide whether the compound forms II or I predominantly.
ACCESSION NUMBER: 1964:15788 CAPLUS
DOCUMENT NUMBER: 60:15788
ORIGINAL REFERENCE NO.: 60:2729g-h,2730a-f
TITLE: Reaction of secondary amines with formaldehyde
AUTHOR(S): Fernandez, J. E.; Butler, G. B.
CORPORATE SOURCE: Univ. South Florida, Tampa
SOURCE: Journal of Organic Chemistry (1963), 28(11), 3258-9
CODEN: JOCEAH; ISSN: 0022-3263
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

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AB Aromatic azines oxidized with 1 mol equivalent AcO2H yielded monoxides of the general type Ar2C:NN(O):CAr2 (I). Their chemical behavior was determined by an intermol. O-shift whereby diazo and carbonyl derivs. were formed. The I were readily accessible and stable sources for diazo compds.; the rearrangement was initiated by light, heat, and protons. I with Ph3P or Zn-AcOH yielded the corresponding azines. I were cleaved by 1 mol equivalent AcO2H into N and the basic carbonyl derivative Azines exhibited (with 2 mol equivs. AcO2H) the same behavior, which could be utilized for the conversion of the azines to the corresponding carbonyl derivs. I exhibited 2 bands at about 8 and 6.40-6.45 μ, resp., which were attributed to the O atom of the N → O grouping. P2O5 (40-50 g.) in 300 cc. CHCl3 treated dropwise with cooling during 5 h. with 100-130 g. 40% AcO2H gave a solution of anhydrous AcO2H. The appropriate azine (0.1 mol) in about 200-300 cc. CHCl3 (C6H6, CH2Cl2, or CCl4) treated dropwise with cooling and stirring with 0.1 mol AcO2H-CHCl3, kept 36 h. at room temperature, washed, dried, and evaporated gave the corresponding I. (Ph2C:N)2 gave in this manner 25% Ph2C:NN(O):CPh2 (II), m. 157° (EtOH). Similarly were prepared the following ArCH:NN(O):CHAr (Ar, m.p., and % yield given): Ph (III), 131° (MeOH), 51.3; o-ClC6H4, 132-3° (EtOH), 50.0; p-ClC6H4, 163° (dioxane), 57.8; p-BrC6H4 (at reflux), 178° (CHCl3), 46.1; p-MeOC6H4, 159° (dioxane), 58.1; p-MeC6H4, 144° (EtOH), 39.7; α-thienyl, 150° (aqueous EtOH), 57.8; α-furyl, 181° (cyclohexane), 59.3; α-pyrryl, 182° (aqueous EtOH), 64.4. II (1.9 g.) in 100 cc. C6H6 irradiated 5 h. with an immersed UV lamp and distilled gave 0.76 g. BzPh, m. 48°. III (2.24 g.) in 110 cc. C6H6 gave similarly 84% BzH and 0.1 g. unchanged III. III (9.0 g.) heated slowly to 135° (2-3 min.) gave 3.5 g. BzH and 0.6 g. unchanged III. III (4.5 g.) in 50 cc. p-xylene refluxed 4 h. yielded 1.9 g. BzH and 0.4 g. III. III (4.5 g.) in 75 cc. Ac2O heated at 130° gave N, 1.85 g. BzH, and 0.2 g. III. III (15.7 g.) in 175 cc. EtOH warmed with 0.1 cc. concentrated H2SO4 gave 6.1 g. BzH and 7.4 g. PhCH2OEt (IV), b18 80°, n20D 1.4960; a similar run with 25 cc. 2N H2SO4 gave 6.0 g. IV and 6.5 g. BzH. III (11.2 g.) in 110 cc. BuOH gave 7.4 g. PhCH2OBu, b14 105-7°, n20D 1.4828, and 4.6 g. BzH. III (8.96 g.) in 100 cc. cyclohexanol containing a few drops concentrated H2SO4 heated to 50° gave 5.8 g. cyclohexyl benzyl ether and 3.3 g. BzH. III (4.5 g.) and 12.0 g. PhOH treated at room temperature with about 0.05 cc. concentrated H2SO4, kept 1 day, treated with dilute aqueous NaOH, and extracted with Et2O gave 2.7 g. PhCH2OPh, m. 39-40°, and 1.7 g. BzH. Picric acid (12 g.) in 35 cc. Me2CO treated at room temperature with 4.5 g. III gave 1.6 g. 2,4,6-(O2N)3C6H2OCH2Ph, m. 143-5° (C6H6), and 1.5 g. BzH. III (4.5 g.) treated 2-3 min. with 15 cc. concentrated HCl gave 100% N, 3.2 g. BzH, and PhCH2Cl. III with 66% HBr gave 81% PhCH2Br. III (4.5 g.) with 25 cc. 50% H2SO4 gave 91% BzH and 1.3 g. PhCH2OH. III (2.24 g.) in 50 cc. AcOH treated with a few drops concentrated H2SO4, HCl, or H3PO4 gave 100% N. III (9.0 g.) in 70 cc. AcOH and a few drops concentrated H2SO4 kept at 20° and worked up in the usual manner gave 66% BzH and 4.8 g. PhCH2OAc, b18 105-7°. p-MeC6H4SO3H (15 g.) in 100 cc. moist Et2O treated with cooling with 4.5 g. III and worked up after 12 h. gave 73% BzH and 4 g. p-MeC6H4SO3CH2Ph (V), m. 58-9.5°. p-MeC6H4SO3H (25 g.) in 60 cc.

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H2O with 4.5 g. III yielded 1.6 g. PhCH2OH, 0.4 g. V, and 80% BzH. III (2.2 g.) and 10 g. Ph2CHCO2H in CHCl3 treated with a few drops concd. H2SO4 and worked up after 24 h. gave 0.9 g. BzH and 1.4 g. Ph2CHCO2CH2Ph, m.34°. III (2.24 g.) in CHCl3 (or C6H6) treated with 10 mmol AcO2H gave 220-30 cc. N, BzH, BzOH, and 1.9-2.1 g. unchanged III. III (2.24 g.) and 4.0 g. Ph3P in 150 cc. EtOH refluxed 1 h. gave 1.3 g. Ph3PO and 1.3 g. (PhCH:N)2 (VI), m. 92°. III (2.2 g.) in 30 cc. AcOH treated under CO2 with 1 cc. satd. aq. KI and 5 cc. HCl and heated did not liberate iodine. III (1.12 g.) in 20 cc. AcOH warmed with 0.5 g. Zn dust and worked up after 24 h. gave 0.45 g. VI. III (4.5 g.) in 60 cc. AcOH heated with excess Zn dust gave PhCH2NH2 (isolated as 0.6 g. HCl salt) and (PhCH2)2NH (isolated as 2.9 g. HCl salt). III (2.24 g.) in 250 cc. MeOH hydrogenated over 7 g. Raney Ni gave PhCH2NH2 (isolated as 1.9 g. HCl salt) and (PhCH2)2NH (isolated as 0.3 g. HCl salt); the same result was obtained similarly with VI. The appropriate arom. azine (0.1 mol) in 200-300 cc. CHCl3 treated with stirring and cooling with 38 g. 40% AcO2H gave the corresponding arom. aldehyde, ArCHO; in this manner the following (ArCH:N)2 were cleaved (Ar, % yield of ArCHO, and % yield of ArCO2H given): Ph, 80, 9; o-ClC6H4, 84, 6; m-ClC6H4, 79, 5; p-ClC6H4, 86, 6, p-MeOC6H4, 85, 7; p-HOC6H4, 75, -; p-MeC6H4, 85, 10. (PhMeC:N)2 and (p-MeC6H4MeC:N)2 gave similarly 88% PhAc and 91% p-MeC6H4Ac, resp. The appropriate aliph. azine, (RR'C:N)2, (0.1 mol) treated with cooling with 0.2 mol 40% AcO2H gave the corresponding RR'CO and peroxide (R, R', % yield of ketone, and % yield of peroxide given): Me, Me, 30, 30 (trimeric, m. 97°); Me, Et, 28, 18 (and a compd. m. 113°); Et, Et, 82, 15 (and a compd. m. 127°); Me, iso-Pr, 18, 12 (and a compd. m. 115°); Me, iso-Bu, 25, 11 (and a compd. m. 140°). Cyclohexylideneazine gave similarly 22% cyclohexanone and a compd., m. 171°.
ACCESSION NUMBER: 1961:48504 CAPLUS
DOCUMENT NUMBER: 55:48504
ORIGINAL REFERENCE NO.: 55:9330b-i,9331a-d
TITLE: Azine monoxides, preparation and properties
AUTHOR(S): Horner, Leopold; Kirmse, Wolfgang; Fernekess, Hans
CORPORATE SOURCE: Univ. Mainz, Germany
SOURCE: Chemische Berichte (1961), 94, 279-90
CODEN: CHBEAM; ISSN: 0009-2940
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
OTHER SOURCE(S): CASREACT 55:48504

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AB The replacement of an amino or alkylamino group by a hydrazine or alkyl hydrazine moiety in a variety of aralkylamines has yielded a group of potent central stimulants which produce their effect by a dual mechanism: (1) direct stimulation of the central nervous system (analeptic action), and (2) powerful inhibition of the enzyme monoamine oxidase which is responsible for the metabolic destruction of endogenous central excitatory hormones. Structure-activity relations are established and discussed. N-Aminoamphetamine displayed 40 times the monoamine oxidase inhibitory potency of iproniazid (Marsilid). The synthesis of the aralkyl hydrazines was accomplished by the reductive hydrazinolysis of phenylalkanones or reaction of hydrazine with a phenylalkyl halide. It is demonstrated that the Raney Ni cleavage of substituted hydrazines constitutes a convenient means of obtaining pure primary and secondary amines.
ACCESSION NUMBER: 1960:70086 CAPLUS
DOCUMENT NUMBER: 54:70086
ORIGINAL REFERENCE NO.: 54:13447i,13448a-b
TITLE: Central stimulants-chemistry and structure activity relations of aralkyl hydrazines
AUTHOR(S): Biel, John H.; Drukker, Alexander E.; Mitchell, Thomas F.; Sprengeler, Edwin P.; Nuhfer, Patrick A.; Conway, Alvin C.; Horita, A.
CORPORATE SOURCE: Lakesides Labs., Inc., Milwaukee, WI
SOURCE: Journal of the American Chemical Society (1959), 81, 2805-13
CODEN: JACSAT; ISSN: 0002-7863
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
OTHER SOURCE(S): CASREACT 54:70086

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AB Water and several organic liquids form stable and finite contact angles on films of amylose acetate, propionate, butyrate, caproate, and benzoate, and also on films of Me and Et amylose. A plot of the cosines of the contact angles on each polymer against the surface tensions of the liquids yielded characteristic lines somewhat curved and involving 2 linear relations, one for each main class of liquid. Hysteresis effects were pronounced (10-30°) and there existed 2 characteristic lines for each polymer. The wettabilities of the same derivs. of amylose, amylopectin, and cellulose were indistinguishable and established the fact that the surface properties were predominantly determined by the functional groups attached to the polymer chains rather than by mol. configurations. The wetting characteristics correlated with the chain lengths of the substituent groups. The angles on the opposite surfaces of films of amylose butyrate and ethyl amylose were very little different for films stripped from substrates of Mylar, Kel-F, and Teflon, but the angles were much lower and less reproducible on surfaces stripped from Hg. Induced orientation was postulated.

ACCESSION NUMBER: 1959:14764 CAPLUS
DOCUMENT NUMBER: 53:14764
ORIGINAL REFERENCE NO.: 53:2739f-h
TITLE: Wetting of polymer surfaces. II. Contact angles of liquids on esters and ethers of amylose and amylopectin
AUTHOR(S): Scholz, J. J.; Roger, Ray B.; Anderson, J. R.
CORPORATE SOURCE: Univ. of Illinois, Urbana
SOURCE: Journal of Physical Chemistry (1958), 62, 1227-30
CODEN: JPCHAX; ISSN: 0022-3654
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

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AB The contact angles of water and organic liquids were measured on films. OH-containing liquids tended to form unstable angles, with complications due to sorption and swelling effects; however, the initial advancing contact angles of water on starch and cellulose films were finite, ranging from 83 to 15° depending on the degree of prior equilibration. Only on starch was a stable finite water contact angle (of 40°) found. Raw cotton fibers were very hydrophobic and the impurities responsible were progressively removed by solvents and alkali. A number of organic liquids, mainly of halogenated type, formed stable, finite, and reproducible contact angles on these polymer surfaces. Linear relations held between the cosines of the contact angles and the surface tensions of the respective liquids. Each of the polymers possessed a characteristic line and the several lines extrapolated to critical surface tensions between 35 and 42 dynes/cm. The relative positions of these lines suggested that the wettabilities, and free surface energies, of the polymers increase in the order starch, amylopectin, amylose, poly(vinyl alc.), cellulose. In contrast to some other types of polymers, small, or negligible, hysteresis effects were found. Films were prepared by casting from solns. onto various substrates and stripping off. The wetting characteristics of the air sides and the substrate sides of these foils were significantly different, with the effects being most pronounced for amylose and least for poly(vinyl alc.). Induced orientation was postulated and the polar-inducing order of substrates was glass, Hg, Lucite, Mylar, polystyrene, air, Kel-F, and Teflon.

ACCESSION NUMBER: 1959:14763 CAPLUS
DOCUMENT NUMBER: 53:14763
ORIGINAL REFERENCE NO.: 53:2739b-f
TITLE: Wetting of polymer surfaces. I. Contact angles of liquids on starch, amylose, amylopectin, cellulose, and poly(vinyl alcohol)
AUTHOR(S): Ray, B. Roger; Anderson, J. R.; Scholz, J. J.
CORPORATE SOURCE: Univ. of Illinois, Urbana
SOURCE: Journal of Physical Chemistry (1958), 62, 1220-7
CODEN: JPCHAX; ISSN: 0022-3654
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

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AB The bodies are treated with substituted NH4 ions derived from aromatic N compds., e.g., N,N-dimethylbenzylamine, dibenzylamine, diphenylguanidine, 1,3-di-o-tolylguanidine, o-dimethylaminomethylphenol, 2-dimethylaminomethyl-4-tert-butylphenol, 2-dimethylaminomethyl - 4 - (1,1,3,3 - tetramethylbutyl)phenol, and 2,4,6-tri(dimethylaminomethyl)phenol.

ACCESSION NUMBER: 1958:107805 CAPLUS
DOCUMENT NUMBER: 52:107805
ORIGINAL REFERENCE NO.: 52:19066c-d
TITLE: Treatment of clays
INVENTOR(S): Brown, Wm. E.; Giacobine, Clifford R.
PATENT ASSIGNEE(S): Gulf Research & Development Co.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2761837		19560904	US	

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AB In the hope that Raney Cu as a hydrogenation catalyst might help to resolve problems of selective reduction, it was prepared with the same care and under similarly varied conditions as Raney Ni. The alloy containing 50% Al, 45% Cu, and 5% Zn was powdered and separated into 170-, 270-, and 325-mesh particles. Catalyst A was prepared according to Fauconneau (C.A. 31, 3217.1). Adding in small portions during 20 min. 30 g. of the alloy of a given mesh to a stirred and refluxed (at a constant temperature) solution of 60 g. pure NaOH in 140 cc. H2O, keeping the mixture at the same temperature 50 min., cooling, decanting the solution, and washing the catalyst with 12-15 l. distilled H2O, twice with 100 cc. alc., and 3 times with 100 cc. Me2CO gives catalyst B, kept under Me2CO. The reductions were carried out in a Parr bomb capable of withstanding 400 atmospheric/sq. cm. at temps. up to 400° with com. electrolytic H from a cylinder under 150 atmospheric The amount of compound to be reduced, its m.p. or b.p., weight of catalyst (and in parentheses the temperature at which it was prepared and its mesh value), H absorbed (from difference between initial and final pressure), time and temperature of heating, product, its m.p. or b.p. and % yield are: 0.33 mole cyclohexene, b. 82.5, 4 g. A (0° and 170), 0.34 mole H, 1 hr., 170-200°, cyclohexane, b. 80°, 100; 0.33 mole anethole, b15 109°, 4 g. A (0° and 170), 0.33 mole H, 40 min., 150-80°, p-MeOC6H4Pr, b12 90°, 100; 0.15 mole PhCH:CHCH2OH, m. 33°, 2 g. A (0° and 170), 0.18 mole H, 1 hr., 170-210°, Ph(CH2)3OH, b32 140°, 100; 0.05 mole anthracene (I) (in 150 cc. PhMe), -, 2 g. B (90° and 325), -, 45 min., 250°, 9,10-dihydroanthracene (II), m. 170°, 100; 0.2 mole I (in 100 cc. PhMe), -, 4 g. B (60° and 270), 0.2 mole H, 2 hrs., 300°, II, -, 100; 0.15 mole phenanthrene, m. 99°, 5 g. B (90° and 325), 0 mole H, 1 hr., 300°, -, -, -; 0.2 mole C10H8, m. 80°, 2 g. A (0° and 170), 0 mole H, 2 hrs., 300°, -, -, -; 0.45 mole AcEt, b. 79°, 2 g. A (0° and 170) (activated by 0.15 cc. 40% NaOH), 0.48 mole H, 40 min., 200-30°, 2-butanol, -, 100; 0.32 mole iso-PrAc, -, 3 g. A (0° and 170) (activated by 0.15 cc. 40% NaOH), 0.3 mole H, 1 hr., 150-70°, iso-PrCH(OH)Me, -, 100; 0.24 mole (iso-Bu)2CO, m. 168°, 3 g. A (0° and 170) (activated by 0.3 cc. 40% NaOH), 0.22 mole H, 2 hrs., 180-200°, (iso-Bu)2CHOH, b. 173°, 95; 0.5 mole PrCHO, b. 75°, 4 g. A (0° and 170) (activated by 0.2 cc. 40% NaOH), 0.51 mole H, 40 min., 200-30°, BuOH, -, 100; 0.5 mole MeCH:CHCHO, b. 101°, 3 g. B (60° and 170) (activated by 0.4 cc. 40% NaOH), 1.04 mole H, 75 min., 180-200°, BuOH, b. 116°, 100; 0.5 mole Me2C:CHAc, b. 130°, 3 g. A (0° and 170) (activated by 0.3 cc. 40% NaOH), 0.98 mole H, 1 hr., 170-200°, iso-PrCH2CH(OH)Me, b. 131°, 95; 0.57 mole cyclohexanone, b. 155°, 3 g. A (0° and 170) (activated by 0.2 cc. 40% NaOH), 0.56 mole H, 40 min., 160-80°, cyclohexanol, b. 159°, 100; 0.33 mole isophorone, b16 93°, 3 g. B (90° and 325), 0.67 mole H, 75 min., 150-70°, 3,3,5-trimethylcyclohexanol, m. 54°, 100; 0.1 mole PhCH:CHAc, m. 39°, 2 g. A (0° and 170), 0.19 mole H, 45 min., 180-210°, Ph(CH2)2CH(OH)Me, b18 127°, 90; 0.042 mole (PhCH:CH)2CO, m. 112°, 2 g. A (0° and 170) (activated by 0.2 cc. 40% NaOH), 0.11 mole H, 1 hr., 170-200°, [Ph(CH2)2]2CHOH, m. 44°, 90; 0.23 mole BzH, -, 2 g. A (0° and 170), 0.3 or 0.47 mole H, 1 hr., 150-75° or 200-30°, 90% PhCH2OH and 5% PhMe or PhMe, -, -; 0.05 or 0.2 mole, PhAc, b18 92°, 3 g. A (0°

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and 170) (activated by 0.2 cc. 40% NaOH) or 3 g. B (50° and 325), 0.6 or 0.39 mole H, 1 hr., 150-75° or 200-40°, PhEt and PhCH(OH)Me, or PhEt, -, -, -, 10, 85, and 100; 0.11 or 0.5 mole, Ph2CO, m. 48°, 3 g. B (50° and 170 or 60° and 270), 0.1 or 1.2 mole H, 1 hr., 150° or 230-50°, Ph2CHOH or Ph2CH2, -, -, 95 and 100; 0.1 mole benzoin, -, 3 g. A (0° and 170) (activated by 0.2 cc. 40% NaOH), 0.11 or 0.29 mole H, 1 hr. or 20 min., 150° or 250°, [PhCH(OH)]2 or (PhCH2)2, -, -, 95 and 100; 0.2 mole RCH:CHBz (R = 2-furyl) (in 100 cc. MeOH), 2 or 3 g., B (50° and 325) (activated by 0.15 cc. 40% NaOH), 0.5 or 0.6 mole H, 80 min., 160-80° or 200-30°, R(CH2)2CH(OH)Ph, b2 100° or R(CH2)3Ph, b2 100°, 95 or 100; 0.58 mole 2-furaldehyde, b15 54°, 3 g. A (0° and 270) or B (50° and 325), (activated by 0.2 cc. 40% NaOH), 0.62 or 1.01 moles H, 50 min. or 2 hrs., 150-65° or 200-40°, furfuryl alc. (III), b15 68°, or III and 2-methylfuran, 95, or 20 and 70; 0.3 mole PhCN, -, 2 g. A (0° and 170), 0.58 mole H, 1 hr., 180°, PhCH2NH2, -, and (PhCH2)2NH, -, 40 and 45; 0.1 mole coumarin (in 50 cc. MeOH), 1 g. A (0° and 170) or 2 g. B (60° and 170), 0.07 or 0.3 mole H, 45 min. or 2 hrs., 140-60° or 210-40°, hydrocoumarin, b20 165°, or o-HOC6H4CH:CHCH2OH, benzoate, m. 98°, 100 or 90; 0.1 mole 2-naphthol, -, 2 g. B (50° and 170) (activated by 0.2 cc. NaOH), 0.18 mole H, 2 hrs., 260-80°, tetrahydro-2-naphthol, b3 117°, 80; 0.5 mole 1-naphthol, 3 g. A (0° and 170) (activated by 0.2 cc. 40% NaOH), 0.24 mole H, 80 min., 270-90°, 1,2,3,4-tetrahydronaphthalene, b. 204°, 40; 0.2 or 0.1 or 0.1 mole PhNO2, 3 g. B (60° and 270) or (90° and 325) (activated by 0.3 cc. NaOH) or (90° and 325) (activated by 0.3 cc. Et3N), 0.6 or 0.27 or 0.27 mole H, 90 or 45 or 45 min., 170° or 140° or 140°, PhNH2 in all 3 cases, 100 in all cases; 0.072 mole p-O2NC6H4NH2 (in 50 cc. C6H6), 1 g. B (90° and 325) (activated by 0.4 cc. Et3N), 0.24 mole H, 15 min., 240-60°, p-C6H4(NH2)2, 100; 0.036 mole o-O2NC6H4NH2 (in 50 cc. C6H6), 1 g. B (70° and 270°) (activated by 0.2 cc. Et3N), -, 10 min., 220-50°, o-C6H4(NH2)2, -, 95; 0.072 mole m-O2NC6H4NH2 (in 25 cc. C6H6), 1 g. B (90° and 325) (activated by 0.4 cc. Et3N), 0.25 mole H, 15 min., 230-50°, m-C6H4(NH2)2, -, -; 0.15 mole p-O2NC6H4OH (in 50 cc. C6H6), 3 g. B (70° and 325) (activated by 0.3 cc. 40% NaOH), 0.42 mole H, -, -, p-HZNC6H4OH, -, 100; 0.05 mole m-C6H4(NO2)2 (in 25 cc. C6H6), 1 g. B (90° and 325) (activated by 0.2 cc. 40% NaOH or 0.2 cc. Et3N), 0.19 or 0.36 mole H, 30 or 15 min., 180-200° or 250°, m-O2NC6H4NH2 or m-C6H4(NH2)2, -, -, -, -; 0.025 mole o-C6H4(NO2)2 (in 50 cc. PhMe), 1 g. B (60° and 325) (activated by 0.2 cc. Et3N), 0.02 mole H, 35 or 25 min., 200° or 245°, o-O2NC6H4NH2 or o-C6H4(NH2)2, -, -, -, -; 0.025 mole p-C6H4(NO2)2 (in 50 cc. PhMe), 1 g. B (60° and 325) (activated by 0.2 cc. Et3N), 0.02 mole H, 30 or 40 min., 200° or 250°, p-O2NC6H4NH2 or p-C6H4(NH2)2, -, -, -, -. Thus hydrogenation in the presence of Raney Cu can be applied to the selective reduction of many types of compds. contg. several reducible groups.

ACCESSION NUMBER: 1956:88889 CAPLUS
DOCUMENT NUMBER: 50:88889
ORIGINAL REFERENCE NO.: 50:16651h-i,16652f-i,16653a-i
TITLE: Catalytic hydrogenation in the presence of Raney copper
AUTHOR(S): Jadot, J.; Braine, R.
CORPORATE SOURCE: Univ. Liege, Belg.
SOURCE: Bull. soc. roy. sci. Liege (1956), 25, 62-78
DOCUMENT TYPE: Journal

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LANGUAGE: Unavailable

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AB cf. C.A. 49, 13261h. The influence of ring size, conjugation, and functional groups on the enamine-imine tautomerism of some cyclic and open unsatd. organic bases has been investigated by spectrophotometric methods. Most secondary or primary vinylamines described in the literature appear to be imines. Hexahydroindole (I) (50 mg.) in dry Et2O treated with 0.8 equivalent 0.1N HCl in Et2O, and the colorless precipitate washed with Et2O and recrystd. from CHCl3EtOAc gave I.HCl, very hygroscopic crystals, m. 160-2° (all m.ps. are corrected). Cyclohexanone anil (II), b0.2 79° treated with HCl in Et2O and the crystalline precipitate washed with Et2O gave II.HCl.0.5H2O, colorless rods, m. 131-3°, with bubbling (sublimed above 100°); attempted recrystn. from EtOH-Et2O gave PhNH2.HCl, m. 198°. Cyclohexylidene-aniline (88 g.), b0.3 78°, treated 15 h. with a lively stream of O at 80° and the mixture extracted with Et2O, C6H6, and MeOH left compound C18H20N2O2 (III), rectangular prisms, m. 239-40° (from MeOH). The oxidation mixture of another run digested with warm CHCl3, the dark solution extracted with saturated aqueous NaHCO3, the extract acidified with AcOH and extracted with Et2O, and the extract worked up gave 0.28 g. acidic fraction; the CHCl3 solution extracted with 2N alkali and the extract neutralized with AcOH and extracted with Et2O gave a phenolic fraction (0.49 g.), light brown viscous liquid, which darkened in air; the CHCl3 solution extracted with 2N HCl, and the extract adjusted to pH 6 to give PhNH2 and then adjusted to pH 8 gave strongly basic material C23H30N2O3, plates, m. 157-9° (from MeOH); the residual CHCl3 extract evaporated to dryness and the C6H6-soluble part of the residue chromatographed on Al2O3 with hexane gave a compound C18H16N2, large colorless plates, m. 109-10.5° (from pentane); the C6H6-insol. part of the neutral fraction gave more III, m. 239-40°. Et β-aminocrotonate (IV) in Et2O treated with HCl in Et2O gave MeC(:NH)CH2CO2Et, crystalline powder. 2-Carbethoxycyclopentanone treated with dry NH3 gave 2-carbethoxycyclopentenylamine (V), colorless plates, m. 59° (from petr. ether). V in Et2O treated with picric acid (VI) in Et2O or with HCl gave NH4 picrate or NH4Cl. 2-Carbethoxycyclohexanone treated with dry NH3 gave Et tetrahydroanthranilate (VII), colorless scales, m. 75°; it gave with VI or HCl in Et2O the NH4 salts. Hydratropic aldehyde (VIII) (2 g.) in 10 cc. MeOH saturated at 0° with dry NH3 and kept 4 days at -5° yielded 1.7 g. MePhCHCH:NH (IX), colorless rectangular prisms, m. 98-105°. MeOH saturated at -5° with NH3 added to VIII in MeOH and kept overnight gave IX, microcryst. powder, m. 100-5° (from EtOH). VIII in EtOAc treated with NH3 with or without cooling gave hexagonal prisms, m. 96-8° (clear at 102°). IV, V, VII, and IX showed 1 single NH band at 3.05, a very sharp and strong C:NH band at 6.02, and bands at 6.24, 6.70, 6.89, 7.28 (C-CH3). The addition of 0.1N CCl3CO2H in CHCl3 to the imine in CHCl3 gave a C:O band at 5.82, but no ammonium or immonium bands. IX (2.5 g.) refluxed 2 h. with 100 cc. 20% KOH in MeOH gave colorless, hexagonal crystals, m. 135-7° (from hot EtOH). VIII (5 g.) in 10 cc. MeOH saturated at 0° with dry Me2NH, the solution slowly evaporated in a vacuum desiccator, the residue digested with 5-cc. portions petr. ether in the cold, and the exts. kept in the cold room gave a compound C11H15NO2.1/3H 2O (X), sheaves of glistening, colorless, hygroscopic needles, m. 150-2° with crystalline transformation at 112-20°. Excess dry Me2NH passed below 50° through 5 g. VIII and the mixture extracted with Et2O gave some X; the reaction product distilled gave MePhC:CHNMe2 (XI), colorless mobile

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bl 52; it turned yellow in air and light. The IR absorptions of XI are given. XI in Et2O treated with HCl in Et2O gave Me2NH.HCl. XI with VI gave Me2NH picrate. VIII (4.47 g.) treated with 4.0 g. p-MeOC6H4NH2 in 15 cc. MeOH and the cryst. product recrystd. from MeOH gave the p-methoxyanil (XII) of VIII, m. 70-80° (clear, slightly yellow melt at 92°); turned yellow and sticky in air. XII in CHCl3 autoxidized so rapidly that it exhibited the same NH and CO bands as p-MeOC6H4NHCHO (XIII). XII in Et2O or EtOAc shaken under O consumed 1 mol O rapidly; the oxidized soln. strongly liberated iodine during and shortly after the O uptake. The residue from autoxidized solns. (crystals embedded in a slightly yellow oil) triturated with petr. ether in the cold, and the crystals recrystd. from Et2O gave XIII; the petr. ether soln. evapd. and the residue treated with 2,4-(O2N)2C6H3NHNH2 gave 2,4-(O2N)2C6H3NHN:CMePh, m. 248-50°. VIII (4.47 g.) in 20 cc. C6H6 refluxed 0.5 h. with 3.1 g. PhNH2 with azeotropic removal of 0.6 cc. H2O and evapd. in vacuo yielded the anil (XIV) of VIII, long silky, colorless needles, m. 134-6°, which showed a strong and narrow NH band at 2.98, a very strong C:N band at 6.05, very strong Ph at 6.28, and weak C-CH3 at 7.25μ. XIV gave XIV.HCl, irridescent scales, m. 244-8° (sublimed at 150°); sharp NH at 2.98μ in CHCl3, traces of ammonium and immonium bands, strong band at 6.05 in CHCl3 and 5.87μ in Nujol. Ph2CHCHO (4 g.) (from hydrobenzoin) in 10 cc. EtOH satd. at 0° with dry NH3 and kept at -5° gave Ph2CHCH:NH (XV), hard colorless crystals, m. 75-82°; 2 strong bands at 6.02 and 6.10 indicative of a mixt. of imine and enamine. The same reaction in 10 cc. EtOAc gave after 48 h. XV.0.25AcOEt, fine fluffy needles, m. 91°; NH2 at 2.97 and 3.05, CO at 7.75 (EtOAc), strong imine at 6.01, weaker enamine at 6.11. A similar run but in Et2O gave colorless hard pellets, m. 91°; weak band at 6.11; the Et2O mother liquor kept 3 days at -5° gave colorless needles, m. 89°; pure imine 2.97, 3.06 and bonded secondary NH. Solns. of the various XV prepns. in CS2 dild. carefully with petr. ether and kept 24 h. at room temp. gave (Ph2C:CH)2NH, silky colorless needles, m. 148-50°; strong and narrow NH band at 2.98. XV and p-MeOC6H4NH2 (equimol. amts.) warmed in MeOH and allowed to stand deposited the p-methoxyanil (XVI) of XV, silky, hygroscopic, very unstable needles, m. 48° (cloudy) with sintering at 40° and clearing at 75°; sharp and narrow band at 2.97 in CHCl3 and at 2.98 at 5.91μ indicative of XIII formed by rapid autoxidn. XVI (3 g.) in 20 cc. EtOAc absorbed within 20 min. 235 cc. O when agitated under O; the oxidized soln. gave a hydroperoxide test (neg. after 2 h.); the soln. concd. and dild. with petr., ether deposited XIII, rosettes of short rectangular rods, m. 80-2°. Dehydrobufotenine (XVII) picrate (30 mg.) in 3 cc. 2N HCl extd. with EtOAc, and the aq. soln. concd. in a desiccator gave XVII.HCl, long colorless needles, losing their transparency above 100°, charring at 215°, and progressively darkening and decompg. without melting up to 300°. Free dihydroberberine, prepd. from the yellow-red HCl salt from the filtrate of oxoberberine, recrystd. from C6H6 gave pure material, yellow prisms with a green tinge, m. 157-9° (decompn. starting at 146°).

ACCESSION NUMBER: 1956:77615 CAPLUS
DOCUMENT NUMBER: 50:77615
ORIGINAL REFERENCE NO.: 50:14595i,14596a-i,14597a-d
TITLE: Infrared diagnosis of the hydrochlorides of organic bases. III. Imine-enamine systems and the mechanism of their oxidation
AUTHOR(S): Witkop, Bernhard
CORPORATE SOURCE: Natl. Insts. of Health, Bethesda, MD
SOURCE: Journal of the American Chemical Society (1956), 78,

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2873-82
CODEN: JACSAT; ISSN: 0002-7863
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
OTHER SOURCE(S): CASREACT 50:77615

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AB A number of secondary amines was subjected to the Sommelet reaction. PhCH2NH-Me, PhCH2NHCHMe2, (PhCH2)2NH, (p-O2NC6H4CH2)2NH (I), and (p-MeO2CC6H4CH2)2NH (II) gave the corresponding aromatic aldehyde in 15, 6, 25-30, 31-48, and 12.2% yield, resp. The Sommelet reactions were carried out by refluxing 0.005-0.02 mole of the appropriate amine (or HCl salt) and 0.01-0.04 mole hexamine in 20 cc. 50% AcOH 1 hr. at which time a 2nd. amount of hexamine equal to the 1st was added, refluxing 1 hr., acidifying strongly, boiling, cooling, and extracting with Et2O, and neutralizing the extract with 20% aqueous Na2CO3 and processing. The aldehydes formed were determined by diluting the residue with H2O or EtOH to a solution of 10.0 + 10-5M and measuring the optical density. In the reaction with II, the solid aldehyde was determined as such. BzH (106 g.) treated with vigorous shaking with 110 g. 35% aqueous MeNH2, the mixture refluxed 0.5 hr. and cooled, and the upper layer worked up gave 85.7 g. PhCH: NMe (III), colorless viscous oil, b. 180-1°, nD20 1.5540. III (60 g.) in 125 cc. absolute EtOH hydrogenated at 80° and 100 atmospheric pressure over Raney Ni yielded 37.0 g. PhCH2NHMe (IV), b. 182-8°. Crude IV in 27 cc. concentrated H2SO4 and 81 cc. H2O refluxed 0.5 hr., cooled, washed with Et2O, strongly basified with KOH, and extracted with Et2O yielded pure IV, b. 184-5°, nD20 1.5235. BzH (1.0 mole) and 1.0 mole iso-PrNH2 gave similarly 0.415 mole PhCH2NHCHMe2, b10 93°, nD20.5 1.5020. p-O2NC6H4CH2Cl (51.3 g.) and 300 cc. concentrated NH4OH heated until the resulting oil solidified, the solid filtered off and extracted with 1 l. boiling 1:1 HCl, and the extract cooled deposited 8.3 g. I.HCl, m. 217.5-19°. p-BrCH2C6H4CO2Me (V) was converted by the method of Emerson and Heimsch (C.A. 46, 1391i) to 85.8% II.HBr and this further to II.HCl, m. 254.5-5.5° (corrected) (from boiling H2O). p-MeOC6H4CHO (60 g.) in 100 cc. PhMe refluxed 1.5 hrs. with 48.2 g. PhCH2NH2 and the PhMe removed gave p-MeOC6H4CH:NCH2Ph (VI), white waxy solid, m. 39.9-40.8°, b. 176-81°. VI (88.3 g.) hydrogenated at 100° and 1500 lb. pressure over Raney Ni yielded 50.0 g. p-MeOC6H4CH2NHCH2Ph (VII), b3 170-2°; VII.HCl, m. 214-15°. p-HOC6H4CHO and PhCH2NH2 gave 95.3% p-HOC6H4CH:NCH2Ph (VIII), m. 208-1°. VIII (23.0 g.) in 300 cc. EtOH hydrogenated at 25° and 1500 lb. over Raney Ni, filtered, diluted with 5 vols. H2O, and extracted with Et2O, and the extract saturated with dry HCl yielded 18 g. p-HOC6H4CH2NHCH2Ph (IX).HCl, m. 217-19°. PhCH2NH2 (53.6 g.) and 42.9 g. p-O2NC6H4CH2Cl in 250 cc. EtOH refluxed 4 hrs., diluted with 900 cc. H2O, and extracted with Et2O, the extract evaporated, and the residue treated with boiling 2% HCl gave 29.6 g. p-O2NC6H4CH2NHCH2Ph (X).HCl, m. 248° (decomposition) (from absolute EtOH). PhCH2NH2 and X gave similarly 34.2% p-MeO2CC6H4CH2NHCH2Ph (XI).HCl, m. 233-4°. p-MeOC6H4CH2NH2 (XII) and p-O2NC6H4CH2Cl yielded 31.6% p-O2NC6H4CH2NHCH2C6H4OMe-p (XIIa).HCl, m. 222-3°. XII and V gave 24.6% p-MeO2CC6H4CH2NHCH2C6H4OMe-p (XIII). HCl, m. 245-6°. The Sommelet reaction was carried out with the following amines (% yields of resulting aldehydes given): VII, 51.1 (46.2, 57.1) BzH, 27.6 (23.1, 29.9) p-MeOC6H4CHO; IX, 53.9 (59.2) BzH, 10.8 (8.6) p-HOC6H4CHO; X, 44.9 (46.2, 30.6) p-O2NC6H4CHO, 23.9 (23.2, 12.7) BzH; XI, 36.0 (36.0) p-MeO2CC6H4CHO, 25.5 (24.1) BzH; XIIa, 34.6 p-MeOC6H4CHO, 26.0 p-O2NC6H4CHO; XIII, 29.8 (34.0, 33.7) p-MeOC6H4CHO, 30.7 (30.3, 30.8) p-MeO2CC6H4CHO. Hexamine (18.6 g.) in 175 cc. CHCl3 heated about 5 min.

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with 30.6 g. V yielded 45.4 g. p-carbomethoxybenzylhexaminium bromide (XIV), m. 175° (decompn.). XIV (14.84 g.) in 40 cc. 50% AcOH heated 2.75 hrs., acidified strongly with concd. H2SO4, cooled, and extd. with Et2O, the ext. neutralized with 20% aq. Na2CO3 and evapd., and the crude product (5.4 g.) recrystd. from petr. ether yielded 4.9 g. pure p-MeO2CC6H4CHO, m. 62-3°.
ACCESSION NUMBER: 1956:74082 CAPLUS
DOCUMENT NUMBER: 50:74082
ORIGINAL REFERENCE NO.: 50:13950f-i,13951a-d
TITLE: Some secondary amines in the Sommelet reaction
AUTHOR(S): Snyder, H. R.; Demuth, John R.
CORPORATE SOURCE: Univ. of Illinois, Urbana
SOURCE: Journal of the American Chemical Society (1956), 78, 1981-4
CODEN: JACSAT; ISSN: 0002-7863
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AB The oxidation of VIII with Br in EtOH and with Me3COCl followed by reaction with base has been studied. Two reaction paths are proposed, one to form (PhCH2)2 (XX) by an unusual N evolution and the other for the formation of a tetrazene and its decomposition products PhCH2NH2, (PhCH2)2NH (XXI), and BzH. From the Br oxidation, EtOBz was also isolated. From the oxidation with Me3COCl in addition to normal products some (PhCH2)2NNHCH2Ph (XXII) was found. The oxidation of I with KMnO4 was examined and the products compared with the previously reported (C.A. 48, 5119a) Br oxidation of the same compound It is concluded that resonance stabilization of the intermediate after loss of N favors the abnormal reaction, that is the N elimination without tetrazene formation. VIII (42.4 g.) 1200 cc. EtOH, and 600 cc. H2O treated dropwise with 70.4 g. Br, the mixture stirred 21 hrs. at room temperature (3.047 l. N was evolved after 3 hrs.), the mixture concentrated to 800 cc., and the crystalline deposit filtered off gave 14 g. XXI, m. 265-6° (from EtOH-Et2O) (all m. ps. are corrected); the acidic filtrate diluted with 1.4 l. H2O and extracted 10 times with Et2O, the extract washed neutral with H2O, dried, and evaporated, the residue distilled, the white solid deposit (in the condenser) dissolved in Et2O, washed with 5% aqueous KOH, H2O, aqueous NaHSO3, and H2O, dried, and evaporated, and the residue (3.3 g.) recrystd. from aqueous EtOH gave XX, m. 52-3°; the liquid fraction of the distillate treated with 40% aqueous NaHSO3 and extracted 3 times with Et2O, the extract washed again twice with 40% aqueous NaHSO3, and the addition product (10.4 g.) decomposed gave BzH (2,4-dinitrophenylhydrazone, m. 234-6°); the Et2O extract from the aqueous NaHSO3 phase dried and evaporated, and the residue fractionated several times gave 2.44 g. slightly impure EtOBz, b3.25 64.5-67°, nD26 1.5090. The Et2O-extracted aqueous acidic layer cooled, basified strongly with solid KOH, and extracted 5 times with Et2O, and the extract dried and fractionated gave 5.1 g. PhCH2NH2, b1.3 36-8°, b1.75 42°, nD25.5 1.5385 [picrate, m. 196-8° (decomposition)], and 3.4 g. XXI, b0.6 102°, nD25.5 1.5720 (picrate, m. 91-3°). I oxidized in the usual manner with KMnO4, but the Et2O solution of the mixture chromatographed on Al2O3 with dry Et2O gave 1.35 g. mixed cis- and trans-III, m. 161.8-2.8°, followed by 1.3 g. tetrazene of I. VIII (15 g.) in 150 cc. dry Et2O treated carefully dropwise at 0° with 8.08 g. 95% Me3COCl during 15 min., the mixture treated with excess KOH pellets and then 40 cc. absolute EtOH, warmed to room temperature, stirred overnight, and filtered, the filtrate evaporated at room temperature, the residual mixture of oil and solid filtered, the filter residue washed with Et2O, and the extract dried and evaporated gave 2.3 g. tetrabenzyltetrazene, m. 99-100°; the oily filtrate distilled gave 1.05 g. XX, b0.65 85.5°, nD27 1.5581, m. 52-3°; the next fraction of the distillate dissolved in Et2O and filtered, and the filtrate washed with 20% HCl and evaporated gave 0.6 g. XXI.HCl, m. 250-6°; the combined original Et2O solution and the Et2O extract from the aqueous acidic layer dried and evaporated gave 0.7 g. XX, m. 49-52°; the aqueous acidic layer basified gave 0.35 g. dark oil which gave only small amts. impure XXII. In another run separation of the tetrabenzyltetrazene followed by acid and base extraction of the mixture gave a neutral fraction which

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distd. yielded 4.5% XXII, m. 86-7° [picrate, m. 140-1° (decompn.)]. A subsequent fraction of the original distn. dissolved in Et2O and filtered, and the filtrate treated with 20 cc. 25% HCl gave 1.2 g. XXI.HCl; the aq. layer gave an addnl. 2.1 g. XXI.HCl; the Et2O layer dried and evapd., and the solid residue (0.4 g.) recrystd. from EtOH gave trans-stilbene, m. 117-20°. The last fraction of the distn., a light green-yellow oil, dissolved in Et2O treated and with HCl gave a white ppt. of XXI.HCl in the Et2O phase; in another run the oil fractionated gave a distillate, b6 192°; the Et2O ext. evapd. and the residual sweet smelling reddish purple oil treated with 2,4-(O2N)2C6H3NHNH2 gave 2,4-(O2N)2C6H3NHN:CHPh, m. 237-8°; however, the oil distd. gave a solid which could not be purified

ACCESSION NUMBER: 1956:24153 CAPLUS
DOCUMENT NUMBER: 50:24153
ORIGINAL REFERENCE NO.: 50:4935i,4936a-h
TITLE: Azo compounds. XIV. Oxidation studies of 1,1-disubstituted hydrazines
AUTHOR(S): Overberger, C. G.; Marks, Burton S.
CORPORATE SOURCE: Polytech. Inst. of Brooklyn, Brooklyn, NY
SOURCE: Journal of the American Chemical Society (1955), 77, 4104-7
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DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

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GI For diagram(s), see printed CA Issue.
AB The rearrangements of various substituted isoindolinium bromides through the agency of intermediate "ylides" are discussed at length. To 100.8 g. BzH in 300 ml. MeOH at 0° was added dropwise 107.2 g. PhCHZNH2, followed, after standing at room temperature and brief heating, by a 5-hr. hydrogenation with Raney Ni at 45-50°, giving 86% (PhCH2)2NH (I), b0.14 126-8°; Bz derivative, m. 112.0-12.8° (cf. Franzen, C.A. 3, 2562). To 92.4 o-C6H4(CH2Br)2, m. 93-4.5°, in 250 ml. CHCl3 at 0° was slowly added 157.8 g. I in CHCl3, giving 95.1 g. 2,2-dibenzylisoindolinium bromide (II), m. 223.3°-4.5° (from EtOH-AcOEt, 4:1); corresponding iodide, m. 196.5-7.5°. II (9.5 g.) in 10 ml. Et2O with 32.5 ml. 0.83N PhLi in Et2O reacted exothermically, giving (presumably) the corresponding "ylide," which then rearranged to o-C6H4.CH2.NCH2Ph.CHCH2Ph; this, when heated at 100°/0.1 mm., gave PhMe (condensed at -80°). The corresponding still residue in Et2O kept 4 days at room temperature with 3.6 g. MeBr formed 1.8 g.
1,2-dibenzyl-2-methylisoindolinium bromide (III), m. 208.5-9.0° (also formed. but m. 211.2-11.4°, from 1-benzyl-2-methylisoindoline, b0.01 105-8°, and PhCH2Br). The Et2O filtrate from III with 1.96 g. maleic anhydride gave, within 3 days, 0.94 g. (crude) IV, m. 152-2.5° (after trituration with EtOH and crystallization from AcOEt-petr. ether). The filtrate from IV, evaporated, gave 1.89 g. of a
tertiary amine, C2ZH21N, m. 70-70.5° (from MeOH), whose infrared absorption spectrum indicated a Me group, which may have resulted from a Sommelet rearrangement; its structure, while still somewhat uncertain, is probably that shown by 2-benzyl-1-(o-tolyl)isoindoline (V). To 12.5 g. 2,3-C10H6Me2 in 130 ml. dry CCl4 in a quartz vessel was added 28.5 g. purified N-bromosuccinimide mixed with 0.2 g. Bz2O2 and the mixture refluxed and irradiated 40 min. with ultraviolet light, giving 14.5 g. 2,3-C10H6(CH2Br)2 (VI), m. 144.3-5.5° (from CHCl3), 3.1 g. of which in 20 ml. CHCl3 with 1.2 g. Me2NH, kept sealed 48 hrs. at room temperature
and then heated several hrs. at 50°, evaporated, extracted with H2O, and made alkaline, gave 2.2 g. 2,2-dimethyl-5,6-benzisoindolinium bromide (VII), m. 284-4.5° (from EtOH); corresponding iodide, m. 285-6°. VII (3.06 g.) in 5 ml. absolute Et2O under N was stirred with 11 ml. 1.09N PhLi at 18° (and in a series of other expts. at 2°, 15°, and 30°) in a fully described apparatus provided with an electromagnetic stirrer, which could be sealed off, but which also permitted the collection and quant. determination in a gasometer of CH4 evolved
during the reaction. When VII had reacted almost completely, the Et2O solution, which had been brown, returned to yellow, and the CH4 approximated 50% of that theoretically possible (actually 47% when carried out at 18°). This would correspond to a 50% yield each of 2-methyl-5,6-benzisoindole (VIII) and 1,2-dimethyl-5,6-benzisoindoline (IX). Although the presence of VIII was indicated by a pos. Ehrlich test, VIII could never be isolated, nor could any adduct with maleic anhydride be obtained. This failure is ascribed to the extreme sensitivity of VIII to O and acids. On the other hand, 1 g. IX was isolated from the Et2O solution, and after extensive purification, including sublimation at 80-100°/0.01 mm., it m. 91-2° (from Et2O); picrate, m. 187-7.5°; MeBr derivative, m. 240-1° (from BuOH). An Et2O solution of all nonvolatile reaction products (when PhLi reacted at 30° with VII) gave with maleic anhydride the acid maleate of IX, C18H19O4N, m. 170.5-1.0° (from AcOEt), readily reconverted into IX by warming with aqueous NaOH. To 28.3 g. VI in 450 ml. Me2CO at 40° was added

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over a 7-hr. period 15.3 g. piperidine in 450 ml. Me2CO, the mixt. boiled 15 min., and the cooled, filtered product extd. exhaustively with hot Me2CO, giving 21.2 g. 2,2-pentamethylene-5,6-benzisoindolinium bromide (X), m. 299-300° (from EtOH). X (3.2 g.) shaken 8 hrs. at 45° with 11.4 ml. N PhLi in Et2O, followed by evapn., distn. at 130-40/0.01 mm., extn. of the cryst. distillate with Et2O and 18% HCl, and treatment with aq. NaOH gave 18.5% 1,2-pentamethylene-5,6-benzisoindoline (Xa), C17H19N, m. 101-2° (from MeOH after sublimation at 95°/0.01 mm.). From 1,2-C10H6Me2 in CCl4 and N-bromosuccinimide and Bz2O2 was formed 54.7% 1,2-C10H6(CH2Br)2 (XI), m. 148.5-9.5° (from CHCl3), which (as in the synthesis of VII) gave rise to 41% 2,2-dimethyl-4,5-benzisoindolinium bromide (XII), m. 184-5° (decompn.) (from BuOH by addn. of ligroine and cooling to -20°). This reaction also gave various yields of 1,2-C10H6(CH2NMe2)2, b0.01 92-3°, up to 48.8% (when as much as 38 millimoles Me2NH was used in the reaction), in which case only 30% crude XII was formed. At 30°, 2.8 g. XII reacted vigorously, but only partially, with PhLi in Et2O, giving CH4, the excess of XII being extd. with H2O, followed by HBr, and evapn. to dryness, and isolated as a tetraphenyloborate, m. 185-93°. The Et2O layer (in this reaction), extd. 10 times with 0.5N HCl (XIII) and then washed with N Na2CO3 and H2O under N, gave 8% 2-methyl-4,5-benzisoindole, isolated as the maleic anhydride adduct, C17H13O3N, m. 94-5° (from Et2O). The XIII ext. made alk. and extd. with Et2O gave 1 g. 2,1-MeC10H6CH(NMe2)Ph (XIV), m. 115-16° (after distn. at 0.01 mm. and crystn. from MeOH), giving no Ehrlich or Zerevitinov tests; picrate, m. 180.5-1.0°. 1,2-BrC10H6Me, b13 152-3°, (22 g.), was converted into the corresponding 1-Li deriv., which with 10.6 g. recently distd. BzH in 10 ml. Et2O, followed by washing with aq. NaHSO3, evapn., and distn. with superheated steam, gave 16 g. 2,1-MeC10H6CH(OH)Ph (XV), not crystd.; 12.4 g. XV, treated at 0° with 2.1 ml. PBr3 in 50 ml. abs. Et2O, heated 1 hr. at 90°, and decompd. with H2O, gave in the Et2O layer the liquid PhCHBr-analog, which, heated in a sealed tube at 100° with Me2NH in Et2O, gave 2.44 g. XV. N-Methylnaphthalimide (4.2 g.) refluxed 5 hrs. with 0.84 g. LiAlH4 gave, after addn. of H2O, steam distn., extn. of the distillate with Et2O, washing the ext. with aq. HCl, and addn. of aq. NaOH to the aq. acid ext., 2-methyl-2,3-dihydro-1H-benz[de]isoquinoline (XVI), b0.01 93.5-4.9°, m. 59.5-61° (from petr. ether), yielding with MeBr 88% XVI.MeBr, m. 241-1.5°; XVI.MeI, m. 230-31°. By the usual technique, XVI.MeBr and PhLi in Et2O (preferably at -20°) gave 27-31% 1,2-dimethyl-2,3-dihydro-1H-benz[de]isoquinoline, b0.01 95-105°, isolated either as the picrate (XVII), C20H18O7N4, m. 162.5-3.0°, or as the MeBr deriv., m. 195-6°. By-products of this reaction were Ph2 and appreciable amts. of a glassy resin, (C14H15N)n, not volatile when heated 2 hrs. at 150°/0.01 mm., sol. in Et2O and C6H6, forming a MeI deriv., yellow-brown powder, softening at 200°, which, shaken 6 days with Ag2O suspended in aq. MeOH, gave the corresponding dark red resinous hydroxide (?), which, on protracted heating, gave no Me3N. The possible mechanism of this resin formation is discussed in some detail. To bring assurance that XVI was not an acenaphthene deriv., the following reactions were carried out. 1-Bromoacenaphthene (XVIII) (cf. Bachmann and Sheehan, C.A. 35, 1400.8) was heated 3 hrs. with freshly prepd. Me2NH, yielding 44% 1-(dimethylamino)acenaphthene, b0.01 77-81°; picrate, m. 165-6° (not identical with XVII). XVIII with Me3N gave the trimethyl(1-acenaphthenyl)ammonium bromide, m. 208.5-9.5° (not identical with XVI.MeI). XVIII and PhLi in Et2O, kept 4 days at -15°, gave a nearly black soln. which, when treated at -80° with MeOH and distd. into picric acid in Et2O, gave Me2NH picrate, m.

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156-8°. The still residue was a dark red resin insol. in HCl, and probably a polymer of acenaphthylene. 1-Methylindole (1 millimole) with 1.1 millimoles (.tplbond.CCO2Me)2 kept 24 hrs. in 5 cc. abs. Et2O gave an adduct, b0.01 130-40°, whose dipicrate, C36H30O18N8, m. 159.5-60° (from MeOH). Infrared spectra of the following isoindolines are given and discussed: 1-o-tolyl-2-benzyl, 2-(o-methylbenzhydryl), and Xa.

ACCESSION NUMBER: 1955:64771 CAPLUS
DOCUMENT NUMBER: 49:64771
ORIGINAL REFERENCE NO.: 49:12435e-i,12436a-i,12437a-d
TITLE: Formation of benzisoindoles
AUTHOR(S): Wittig, Georg; Ludwig, Heinz
CORPORATE SOURCE: Univ. Tubingen, Germany
SOURCE: Ann. (1954), 589, 55-76
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
OTHER SOURCE(S): CASREACT 49:64771

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AB For the purpose of finding a new method of synthesis of α-amino acids, preliminary expts. on metalation and alkylation of hydantoin (I) and thiohydantoin (II) were carried out. Benzylation of metalated hydantoin, prepared by interacting I with KNH2 or NaNH2 in liquid NH3, with benzyl chloride (III) gave benzylamine, dibenzylamine and unreacted I; in Et2O or in III, it resulted in recovery of I and III. With II in liquid NH3, benzyl mercaptan and a small amount of a substance distilling at 170-90/20 mm. were obtained.
ACCESSION NUMBER: 1955:35751 CAPLUS
DOCUMENT NUMBER: 49:35751
ORIGINAL REFERENCE NO.: 49:6838d-e
TITLE: Organic syntheses in nonaqueous solutions. II. The alkylation of glycine derivatives in liquid ammonia. 1. Benzylation of hydantoin in liquid ammonia
AUTHOR(S): Shimo, Kotaro; Asami, Ryuzo
CORPORATE SOURCE: Tohoku Univ., Sendai
SOURCE: Bull. Chem. Research Inst. Non-Aq. Solns. (1954), 4, 69-73
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

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AB BrCH2CHBrCO2Me (I) and MeCHBrCHBrCO2Me (II) react with PhCH2NH2 in a manner similar to that of α,β-di-Br ketones. On the basis of this analogy, of chemical reactions, and of mol. refraction and infrared spectra, the reaction products obtained are formulated as 1-benzyl-2-ethyleniminecarboxylic acid esters. I (36.9 g.) in 100 cc. dry C6H6 treated with cooling with 16.1 g. PhCH2NH2 and 30.1 g. Et3N in several portions, the mixture refluxed 3 hrs. and filtered, the filtrate washed with H2O, dried with Na2SO4, and evaporated in vacuo, the residual oil
distilled in a high vacuum, and the distillate, b0.2 96-8°, redistd. gave 20.8 g. (74%) 1-benzyl-2-carbomethoxyethylenimine (III), nD25 1.5238, d25 1.1074, MRD52.81, λmaximum 9.2 μ, was slightly acidic to litmus in EtOH, stable in the dark, and did not give a picrate. III (0.4934 g.) in 10 cc. CHCl3 consumed 14 cc. Br in CHCl3 (0.0312 g./cc.). III (5.5 g.) in 100 cc. absolute EtOH and 2 cc. glacial AcOH hydrogenated 2 hrs. at room temperature and 60 lb. pressure over 200 mg. PtO2 gave 2 cc. of a
basic oil, b0.25 91-3°, nD29 1.5117, which on standing several hrs. deposited a small amount of crystals, m. 88-90° (washed with petr. ether). III (2 g.) in 10 cc. dry Me2CO treated, with cooling, with excess HCl in Et2O, the mixture refrigerated overnight, and the precipitate filtered off,
washed with Et2O, and recrystd. from absolute EtOH-Et2O gave a solid, m. 138-40°, having the structure PhCH2NHCH(CH2Cl)CO2Me.HCl or PhCH2NHCH2CHClCO2Me.HCl. I treated with 3 moles PhCH2NH2, the mixture distilled, the dark brown residue extracted with boiling C6H6 to remove the crude
III, and the remaining white crystalline material dissolved in hot glacial AcOH
and precipitated with absolute EtOH gave 1-benzyl-N-benzyl-2-ethyleniminecarboxamide
(IV), m. 252-4°, which did not react with Br in CHCl3 and reduced KMnO4 in glacial AcOH slowly. IV (0.2 g.) refluxed with 10 cc. 6N HCl and 10 cc. glacial AcOH, and the resulting white precipitate recrystd. from glacial
AcOH-Et2O gave a product, m. 207-9°, having the structure PhCH2NHCH2CH(OH)CONHCH2Ph.HCl and (or) PhCH2NHCH(CH2OH)CONHCH2Ph.HCl, insol. in H2O and dilute HNO3, soluble in concentrated HNO3. II and PhCH2NH2 gave
50% 3-Me derivative (V) of III (possibly the trans form), b0.4 91-3°, MRD 57.37, nD25 1.5144, d25, 1.067, λmaximum 7.2 μ, did not give a picrate and reacted in almost neutral EtOH. V (5 g.) and 4.3 g. PhCH2Br refluxed 4 hrs., the resulting precipitate dissolved in hot Me2CO, diluted with a
small amount of Et2O, and the precipitate recrystd. from absolute MeOH gave (PhCH2)2NH,
m. 257-8°. Propylene oxide (7.4 g.) slowly added to 53.5 g. PhCH2NH2 in 150 cc. 95% EtOH, and the mixture heated 2 hrs. at 40-50°, then to the b.p., cooled, let stand 24 hrs. at room temperature, and distilled gave MeCH(OH)CH2NHCH2Ph (VI), b0.2 93-5°, nD27 1.5270. VI (14.5 g.) and 8.2 g. concentrated H2SO4 heated to 250°, and the mixture cooled slowly, ground with 95% EtOH, filtered off, washed several times with EtOH gave VI sulfate. VI sulfate (6 g.) and 2.5 g. NaOH in 18 cc. H2O heated until an exothermic reaction began, the mixture heated after completion of the reaction 0.5 hr. to 100°, and the resulting oil dissolved in dry Et2O, dried with KOH pellets, and distilled gave 1-benzyl-2-methylethylenimine, light yellow oil, b2 58°, nD27 1.5113, λmaximum 7.2 μ.
ACCESSION NUMBER: 1955:1020 CAPLUS

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DOCUMENT NUMBER: 49:1020
ORIGINAL REFERENCE NO.: 49:172a-h
TITLE: The reaction of α,β-dibromo acid esters with benzylamine
AUTHOR(S): Stolberg, Marvin A.; O'Neill, John J.; Wagner-Jauregg, Theodor
CORPORATE SOURCE: Chem. Corps Med. Labs., Army Chem. Center, MD
SOURCE: Journal of the American Chemical Society (1953), 75, 5045-7
CODEN: JACSAT; ISSN: 0002-7863
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

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AB This work was concerned with the effect of hexane and benzene on the polarizations and apparent moments of amines, the changes in moment produced by different alkyl and aryl groups attached to N, and the comparisons of the polarizations of the pure amines with those of amines in solution at infinite dilution and, where possible, in the vapor state. The dipole moment of aniline in solvents is lower than in the vapor state. In most of the 18 amines studied, the effect of the solvent on the moment of the solute was small. Propyl and butylamine show larger moments in all the solvents used than in the vapor state. The moments for alkylamines fall in the order primary > secondary > tertiary with an approx. constant difference existing for the amines studied. This order is reversed for the benzylamines except that the moment of dimethylaniline is slightly less than that of methylaniline. The variation of polarization with change of concentration depends on the type of amine and its dielec. constant
Small, but definite, changes were found in the apparent mol. solution vols. of the amines in different solvents.
ACCESSION NUMBER: 1953:11238 CAPLUS
DOCUMENT NUMBER: 47:11238
ORIGINAL REFERENCE NO.: 47:2002g-i,2003a
TITLE: The dielectric polarization of solutions. I. The polarizations and apparent dipole moments of various primary, secondary, and tertiary amines in solutions in nonpolar solvents and in the liquid state
AUTHOR(S): Cowley, Eric G.
CORPORATE SOURCE: Acton Tech. Coll., Acton, UK
SOURCE: Journal of the Chemical Society, Abstracts (1952) 3557-70
CODEN: JCSAAZ; ISSN: 0590-9791
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

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GI For diagram(s), see printed CA Issue.
AB To 30 g. Ph2C:NNHPh (I) in dry Et2O were added 16 g. 70% HClO4 (II) and 27 g. Ac2O in Et2O, giving 39-40 g. II salt (III) of I, red needles, m. 186° (decomposition) (from glacial AcOH), rapidly and quantitatively hydrolyzed to I and II. When heated 9 hrs. in dry dioxane at 100°, III remained largely unchanged, giving, however, about 2 g. p-C6H4(NH2)2.2H ClO4, dark yellow, identified by conversion into the free base (IV), m. 139°, and its HCl salt. In this and subsequent rearrangements, full details are given for the separation and identification of small amts. of degradation products which in this case included BzPh, PhNHNH2, PhNH2, and NH3. When 6 g. III was heated in 100 cc. boiling PhBr, small amts. of NH4ClO4 and the II salt of IV formed (exploding, without melting between 200 and 300°) (identified by conversion into the di-Ac derivative of IV, did not m. below 290°). An unidentified violet-black amorphous substance (possibly due to oxidation of IV) was also formed. The mechanism of this p-semidine rearrangement with concomitant reduction and oxidation is discussed. p-MeC6H4NHN: CPh2 (cf. Sah and Lei, C.A. 27, 4222) yielded 70% of the II salt (V), C20H18N2.HClO4, dark red needles, m. 162° (decomposition). V heated briefly in PhBr gave resinous products, and small amts. of p-MeC6H4NH2 (identified as the HCl salt, m. 232°), NH3, traces of BzPh, but no 3,4-(H2N)2C6H3Me (showing that no o-semidine rearrangement had occurred). To 20 g. I, 70 cc. Ac2O, and 10 g. dry ZnCl2 were added 10 cc. AcOH and 10 cc. Ac2O, the mixture warmed on a steam bath, cooled, and the filtered product washed with Ac2O and with C6H6 and dried over H2SO4, giving 30 g. of a compound (VI), C21H18ON2.ZnCl2, hygroscopic crystals, m. 214-15° which with MeOH, followed by H2O, gave Ph2C: NNAcPh (VII), m. 90-1° (from cyclohexane, followed by petr. ether), split quantitatively by concentrated HCl into PhBz and (after treatment with aqueous NaOH) PhNAcNH2, m. 119-20° (from cyclohexane). Heating VI 6 hrs. at 200-20° with excess ZnCl2, followed by treatment with MeOH gave 47% of the theoretical amount of BzPh and 30% of approx. equal parts of IV and 2-methylbenzimidazole, m. 166-8° (after sublimation). In another similar experiment, 20 g. VI (heated with 6.5 g. ZnCl2) gave 5 g. BzPh and the same bases, as well as 0.4 g. o-C6H4(NH2)2, m. 98-99°, thus indicating that both p- and o-semidine rearrangements had occurred. PhCMe:NNHPh gave an 80% (crude) yield of the II salt, yellow leaflets with greenish sheen, m. 158° (from 1:1 Et2O-AcOH); this, refluxed 0.25 hr. in PhBr, gave 4.7 g. of a mixture of NH4ClO4 and 2-phenylindole, m. 186° (from ligroine). Heating Ph2CCl2 and HZNNMe2 5 hrs., followed by Et2O extraction, washing with H2O, drying with K2CO3, and addition of II gave 63% of the II salt (VIIa) of Ph2C:NNMe2, colorless, m. 172° (readily hydrolyzed into PhBz and H2NNMe2), and 2 by-products, (Ph2CCl)2, m. 180° (cf. Finkelstein, C.A. 4, 2641), and β-benzopinacolone, m. 181°. VIIa in Me2CO with excess aqueous NaOH gave an oil, which, extracted with Et2O, gave Ph2C:NNMe2, m. 34° (from petr. ether). Molten VIIa (2 g.) heated 1 hr. at 165-170° gave only about 0.25 g. NH4ClO4, and 0.2-0.25 g. of a compound (insol. in aqueous HCl), m. 150-51° (probably 1-methyl-2-phenylisoindole, the analytical data of which were lost during the war and which up to the present has not been resynthesized); much of the original material was recovered as PhBz and MeZNNH2. PhAc and H2NNMe2 gave PhMeC: NNMe2, colorless oil not crystallizing at -15°; II salt (VIII), colorless needles, m. 107° (from

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EtOH), hydrolyzing slowly in moist air. When heated 2-3 hrs. at 160-70°, 60 g. VIII gave about 12.5 g. (N:CPh.CH2.CH2.N+ Me2)ClO4 (IX), m. 213-14° (by extn. with AcOH and crystn. from H2O), 6.9 g. NH4ClO4, 4.6 g. MeNH3ClO4 (isolated as the oxalate, m. 175°), 0.9 g. Me2NNH2HClO4 (isolated as the oxalate, m. 144-145°), 0.4 g. (Me2N.N:CPh.CH2.CH2)ClO4 (free base (X), m. 35-6°), 1.2 g. (Me2N.N:CPh.CH:CH)ClO4 [isolated as the HCl salt, m. 86° (free base, m. 56°; picrate, m. 130-31°)], 0.1 g. BzCH2CH2NH2.MeClO4 (m. 194-97°), and 2.4 g. dihydrodypnone, m. 72° (from MeOH). (Details of these sepns. are given.) PhMeC:NNMe2 (1.85 g.) and 4.2 g. ZnCl2 were heated 1 hr. at 200-20°, cooled, extd. with MeOH, the filtered ext. poured into H2O, and the mixt. filtered and treated with II, giving 0.55 g. VIII. When the above reaction was carried out with 4 (instead of 3) moles ZnCl2, 23% of the theoretical amt. of VIII was formed. The following derivs. were prepd. from VIII in good yields: picrate, m. 142-3° (from EtOH and dioxane); HI salt (XI), colorless leaflets, m. 220-21° (from EtOHAcOEt) (also formed from 1-methyl-3-phenylpyrazoline and MeI). The probable mechanism for the formation of IX (which contains 1 CH2 group more than VIII) is fully discussed. With 15% aq. KOH, 3 g. IX gave BzMe and, after treatment with HCl, fractionation, and addn. of (CO2H)2, the Me2NNH2 oxalate, m. 144-45° (giving a marked m.-p. depression with (MeNH)2 oxalate, m. 132°). XI carefully heated at 220-40° (at 14 mm. pressure) gave 78% X (picrate, m. 132°) (cf. K. von Auwers and Heinke, C.A. 22,422). BzCH:CH2 (0.7 g.) and 0.5 g. Me2NNH2.2HCl stirred 0.5 hr. at 100° extd. with Et2O and alc., and treated with II gave IX. IX was also formed by heating BzCH2CH2NH2.MeClO4 and Me2NNH2.2HCl at 160-70°. The following derivs. of VIII, were prepd: MeI, C11H17N2I (XII), m. 147° (decompn.); picrate of XII, m. 121°; II salt of XII, m. 145°. Dihydrodypnone semicarbazone, m. 165-6°. Me2NCH2CH2Bz (cf. Mannich and Heilner, C.A. 16, 2497) in Et2O reacted violently with MeI, giving a MeI deriv. (XIII), m. 211-12° (readily split by heating with H2O into BzCH:CH2 and Me3NHI). By treatment with excess aq. AgNO3, filtration, and addn. of NaClO4, XIII gave (2-benzoylethyl)trimethylammonium perchlorate, C12H18O5NCl, m. 196-199° (decompn.) (from PhNO2). BzMe and (PhCH2)2NNH2 gave the corresponding hydrazone, C22H22N2, m. 53-54°; II salt (XIV), m. 163-65° (from PhMe). Heated 5 hrs. at 160-70° 2 g. XIV gave the following compds.: BzCH2CH2Ph, m. 70-71°; PhC:N.N(CH2Ph).CPh:CH (XV), m. 113-14°; a compd., C22H19N2Cl, m. 174-75° (not the HCl salt of either the pyrazole or pyrazoline); NH3 and (PhCH2)2NH (isolated as the HCl salt, m. 258-59°). The HCl salt of XV decompd. about 160° giving XV; the HCl salt of the 1-benzyl-3,5-diphenylpyrazoline proved unstable, and decompd. on attempted recrystn. from EtOH. By refluxing 4.3 g. 1-aminopiperidine with 5.8 g. BzMe, followed by treatment with II (at 0° in Et2O), was formed 6 g. PhCMe:NH(ClO4)N.(CH2)4.CH2, m. 124-25° (from dioxane), which, when boiled 1 hr. in PhNO2, followed by extn. with aq. HCl, then with C6H6, and treatment (of the aq. layer) with 40% NaOH (with subsequent, fully described purifications) gave the base, C13H16N2 or C13H14N2 (probably the latter, i.e., N:CPh.CH:C.N.CH2.CH2.CH2), m. 81° (from MeOH); picrate, m. 177°. The above rearrangements (as well as those reported by other investigators) are fully discussed. Thirty-six references.

ACCESSION NUMBER: 1952:2660 CAPLUS
DOCUMENT NUMBER: 46:2660
ORIGINAL REFERENCE NO.: 46:477g-i,478a-i,479a-d
TITLE: Structural rearrangements of hydrazones
AUTHOR(S): Theilacker, Walter; Leichtle, Otto R.

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CORPORATE SOURCE: Tech. Hochschule, Hanover, Germany
SOURCE: Ann. (1951), 572, 121-44
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

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AB cf. Italian pat. 436,808 (June 14, 1948). Crude SiS2 (containing 80% SiS2), which can be readily prepared by direct synthesis, can advantageously take the place of SiCl4 for the preparation of ortho and polysilicic esters, mixed anhydrides of silico carboxylic acids, and substituted amides of silicic acid. Reaction with alcs. and phenols. The reaction SiS2 + 4ROH → Si(OR)4 + 2H2S [cf. Fr.acte.emy, Ann. chim. phys. [3] 38, 314(1852)] is stoichiometrically complete with the calculated proportion of reagents and with excess alc. With deficient alc., particularly at elevated temps., more SiS2 reacts and less H2S is evolved, and the alkyl silicate contains S. The reaction is probably mSiS2 + (m + 1) Si(OR)4 → (RO)3SiS[(RO)2SiS]2m-1Si(OR)3. These O-alkyl thiopolysilicic esters could not be isolated, but the lack of H2S, the formation of high-boiling products, the formation of polymers with excess SiS2 in the absence of water, and the evolution of H2S when these high-boiling products are treated with dilute acids indicate their formation. Thiols are not formed at relatively low temps.; hence a structure with S-alkyl residues is impossible. Anhydrous phenols react like alcs. With water present, alcs. and phenols react thus: (m + 2)SiS2 + (m + 1)H2O + (2m + 6)ROH → (RO)3Si[OSi(OR)2]mOSi(OR)3 + 2(m + 2)H2S. Reaction with carboxylic acids. In an anhydrous medium, the reaction is SiS2 + 4RCO2H → Si(CO2R)4 + 2H2S. This preparation of Si(CO2R)4 compds. is easier than from SiCl4. They hydrolyze immediately in water, with formation of Si(OH)4, and with amines they react thus: Si(CO2R)4 + 4RNH2 → Si(OH)4 + 4RCONHR. When heated they decompose: Si(CO2R)4 → 2(RCO)2O + SiO2; this offers a method of preparation of anhydrides. More gradual pyrolysis gives intermediate products: mSi(CO2R)4 → (RCO2)3SiO[(CO2R)2O]m-2Si(CO2R)3 + (m - 1)(RCO)2O. With alcs., Si(CO2R)4 compds. react thus: Si(CO2R)4 + 4ROH → Si(OR)4 + 4RCO2H. Reaction with amines. SiS2 reacts with aliphatic and aromatic amines analogously to its reaction with alcs. and acids, but more slowly, and in some cases only at elevated temps. An inert solvent facilitates the reaction. With cold aliphatic amines, the H2S is taken up by the amine: 6RNH2 + SiS2 → Si(NHR)4. Hot primary amines give polymeric imines. In general it is preferable to prepare the amines from SiCl4 rather than from SiS2. Anhydrous MeOH (2000 g.), added very slowly to 1150 g. crude SiS2 (80%) and fractionated, yields 450 g. MeOH, a few cc. of intermediate fraction, 1390 g. Si(OMe)4, and 250-70 g. residue. Similarly, but with distillation in vacuo, 2050 g. EtOH and 1150 g. crude SiS2 yield 1800-1850 g. Si(OEt)4. Distillation can be avoided; e.g., 2200 g. EtOH and 1150 g. SiS2, allowed to react, filtered under pressure or in vacuo, washed with 300 g. anhydrous EtOH, and heated gradually up to 150°, leave 1850 g. Si(OEt)4. Et polysilicates can be prepared not only by hydrolysis of Si(OEt)4, but also by the reaction 5SiS2 + 12EtOH + 4H2O → (EtO)3Si[OSi(OEt)2]3OSi(OEt)3 + 10H2S. E.g., 1435 g. 95% EtOH, added slowly to 1150 g. very cold crude SiS2, refluxed 3 h., filtered cold under pressure, the residue washed with 200 g. 90% EtOH, and the combined filtrates heated at 150° to remove EtOH, yields 1350 g. Et polysilicate. Crude SiS2 (115 g.) and 170 g. anhydrous EtOH, heated 6 h. at 100-120°, filtered in vacuo, the residue (26 g.) washed with Et2O, and the filtrate distilled in vacuo, yield 80 g. Si(OEt)4 and a residue which at higher temps. evolves S compds., including EtSH, and which contains thiosilicates. Crude SiS2 (115 g.) and 380 g. PhOH react violently; the product, heated 1 h. at 180°, cooled, 100 cc. C6H6 added, filtered,

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washed with hot C6H6, and the filtrate distd. in vacuo (6 mm.), yields 8-10 g. PhOH and 300 g. Si(OPh)4. Similarly 115 g. SiS2 and 430 g. con. cresol yield 340 g. tolyl orthosilicate (mixt. of isomers), thick refractive liq., hydrolyzes in moist air. SiS2 (115 g.) and 480 g. mixed xylenols yield 380 g. of xylyl orthosilicate. SiS2 (67 g.) and 325 g. o-dichlorophenol yield after purifn. by CCl4 o-dichlorophenyl silicate, b7 380-400°, m. 63°.
Glacial AcOH (750 g. contg. 1% Ac2O), added to 115 g. SiS2 (keeping the temp. below 60°), heated at 70°, filtered hot, washed with hot glacial AcOH, the filtrate distd. in vacuo (15 mm.) until the temp. reaches 40°, filtered in vacuo, the residue washed with Ac2O at -10° and purified from CHCl3 or ligroin, yields approx. 200 g. Si(OAc)4. The report of Friedel and Ladenburg [Ann. 145, 174(1868)] that it distils unde-compd. applies only to a high vacuum; otherwise it decomp., even in soln. above 50°, according to the reaction Si(OAc)4 → SiO2 + 2Ac2O. It is an energetic acetylating agent; e.g., 5 g. Si(OAc)4 in 30 cc. anhyd. C6H6 and 5 g. PhNH2 in 30 cc. C6H6, added to 100 cc. 5% NaOH at 0°, yields 6.5 g. PhNHAc.
Si(OAc)4 (10 g.), added cautiously to 100 cc. ice-water, the soln. divided into 3 parts, and 5 cc. N NaOAc added to 1 part, 1 cc. N HCl to another part, and nothing to the 3rd part, gives gelatinous solns. at 18° in 5, 11, and 20-22 days, resp. Operating as in the prepn. of Si(OAc)4, but distg. in vacuo above 50°, yields uncrystallizable compds. of the general compn. [SiO(OAc)2]n, which in hydrolysis and acetylation reactions behave like Si(OAc)4. BzOH (186 g.), added in portions to 58 g. SiS2 in 1000 cc. C6H6, refluxed until H2S is no longer evolved, filtered hot, the filtrate evapd. in vacuo, filtered cold in vacuo, and the residue washed with petr. ether, yields Si benzoate, Si(OBz)4, m. 85-90°, hydrolyzes in water to Si(OH)4 and BzOH. Si(OBz)4 (13 g.) in 50 cc. C6H6 and 9 g. PhNH2 in C6H6, allowed to stand 2 h. and poured into 100 cc. ice-cold 5% NaOH, ppt. 17-18 g. PhNHBz. Sublimed (90%) SiS2 (10 g.) and 38 g. ClCH2CO2H, heated 2 h. at 40°, filtered hot in vacuo, and the filtrate allowed to stand, ppts. 5 g. of Si chloroacetate, Si(OCOCH2Cl)4, m. 154° (decompn.). The residue from the original filtration, distd. in vacuo at 50-60°, decomp., yielding 20 g. (ClCH2CO)2O. Si(OCOCH2Cl)4 (4 g.) in 25 cc. C6H6 and 3.5 g. PhNH2, heated 5 min. at 40° and poured into ice-cold 5% Na2CO3, ppt. 7 g. PhNH2.ClCH2CO2H.
SiS2 (10 g. of 90% purity) and 100 g. stearic acid in 200 cc. C6H6, refluxed a long time, filtered, and the filtrate chilled, ppt. Si stearate, Si(OCOC17H35)4, wax, m. 75°. Sublimed (97-8%) SiS2 (15 g.), 40 g. PhNH2, and 100 cc. CCl4, refluxed 6-8 h., filtered, 0.5 of the CCl4 evapd., and allowed to stand, ppt. 0.1-0.2 g. of a compd. contg. 3.20% SiO2 and 15.3% N, perhaps the addn. compd., Si(NHPh)4.14PhNH2. The soln., evapd. to 0.5 its vol., yields Si(NHPh)4, m. 136°. It is formed also from 15 g. 97% SiS2, 40 g. PhNH2, and no solvent by allowing to stand 1 h. at not over 70°, dilg. with hot CCl4, filtering hot, and allowing to stand. (PhCH2)2NH (30 g.) and 5 g. 96% SiS2 in 100 cc. C6H6, refluxed 6 h., filtered hot, and allowed to stand, ppt. Si[N(CH2Ph)2]4, gray. Although SiS2 reacts energetically with primary and secondary aliph. amines, with evolution of H2S, the prepn. of amides by this method is unsatisfactory, because of the formation of sulfides and polysulfides of the amines, which are difficult to eliminate. Comparative expts. with Et2NH and (iso-Pr)2NH showed that SiCl4 is preferable to SiS2. Although SiS2 reacts energetically with OH compds. and with other compds. contg. mobile H, this is not true, contrary to data in the literature, of other classes of org. and inorg. compds., e.g., it does not react with Cl, Br, or I, either dry or in CCl4. Nor does SiS2 react with esters or ketones, contrary to Fr.acte.emy (loc. cit.). Unlike P4S3, P2S5, and CS2, SiS2 does not react with Grignard reagents at the b.p. of Et2O or C6H6.

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ACCESSION NUMBER: 1949:24962 CAPLUS
DOCUMENT NUMBER: 43:24962
ORIGINAL REFERENCE NO.: 43:4630e-i,4631a-i,4632a-c
TITLE: Organic derivatives of silicic acid from silicon disulfide
AUTHOR(S): Malatesta, Lamberto
SOURCE: Gazzetta Chimica Italiana (1948), 78, 753-63
CODEN: GCITA9; ISSN: 0016-5603
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

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AB Because of the value of the preparative method of the catalytic removal of the N-CH2Ph group, a study has been made of the influence of the residue on the N atom upon catalytic debenzylation. All hydrogenations were carried out at room temperature and atmospheric pressure in EtOH or AcOH, using PdO or PtO2 as catalyst. PhCH2NH2, (PhCH2)2NH and PhCH2NHMe are unchanged in the presence of PdO. (PhCH2)3N in AcOH (PdO) or its HCl salt in H2O (PdO) gives 97% of (PhCH2)2NH.HCl; the amine is not reduced by Na and EtOH. Methylcetylbenzylamine in AcOH (PdO) gives 92% of cetylmethylamine-HCl and lauryldibenzylamine gives laurylmethylamine. Dodecyldibenzylamine in AcOH (PtO2) gives 84% of dodecylhexahydrobenzylamine-HCl, m. 218°. (PhCH2)2NNH2 in absolute EtOH (PdO) yields 88% of PhCH2NHNH2; tetrabenzyltetrazene {(PhCH2)2NN:}2 gives (PhCH2)2NH. (PhCH2)3MeNOH with PdO in EtOH readily yields PhCH2NHMe (flavianate, m. 190°; picrolonate, m. 210°), whereas (PhCH2)3MeNI is not reduced. PhCH2NPhMe2Cl gives 90% of cyclohexyldimethylamine. 2-Benzyldihydroisoindole in EtOH (PdO) yields 75% of 1,3-dihydroisoindole, b3 100°. 1,4-Dibenzylpiperazine in AcOH (PdO) gives 92% of piperazine diacetate, m. 234°. α-Monobenzylaminotetrazole gives aminotetrazole. PhCH2NH2 (1 mol.) in AcOEt is treated with a concentrated aqueous solution of 4 mols. of KCN and then dropwise with 1.1 mols. of Br in AcOEt at 5-10°, and the AcOEt solution shaken with 30% NaOH; the alkali removes the benzylcyanamide, which is polymerized to tribenzylisomelamine (1,3,5-tribenzyl-2,4,6-triimino-1,3,5-triazine) (I), m. 129-30°; short heating with HCl gives NH3; with H and PdO in EtOH this yields melamine. The elimination of PhCH2 from 2-imino-1-benzyl-1,2-dihydropyridine is slow and incomplete and is accompanied by nuclear hydrogenation, the products being 2-amino-3,4,5,6-tetrahydropyridine and 2-imino-1-benzylpiperidine (picrate, m. 106°). 2-Benzylaminopyridine does not lose PhCH2 but is hydrogenated to 2-benzylamino-3,4,5,6-tetrahydropyridine, m. 40-1° (picrate, yellow, m. 131°; picrolonate, yellow, m. 199°). Aromatic rings, CO2H and CN groups activate the compds. so that PhCH2 is removed from a sec-N atom. PhNHCH2Ph in EtOH (PdO) gives 97.5% of PhNH2 and PhMe, whereas PtO2 gives mainly cyclohexylhexahydrobenzylamine and small amts. of cyclohexylamine and hexahydrotoluene. PhN(CH2Ph)2 with PdO in EtOH gives 89% of PhNH2 and PhMe. 2-(Dibenzylamino)naphthalene in AcOH (PdO) gives 88% of 2-C10H7NH2 and PhMe. ClCH2CO2H (9 g.) and 40 g. (PhCH2)2NH in 20 cc. dioxane, heated 5 h. at 120°, give 82% of N,N-dibenzylglycocoll, m. 200°; Me ester, m. 41°; hydrogenation in AcOH (PdO) or in EtOH (PdO) gives NH2CO2H (95%) or its Me ester (96%). (PhCH2)2NCN yields NCNH2 or I because of polymerization of PhCH2NHCN if hydrogenation is interrupted before it is complete. (CONHCH2Ph)2 and N,N-dibenzylurethane, b2 169°, b4 181° (82% yield), are stable toward H.
ACCESSION NUMBER: 1943:19038 CAPLUS
DOCUMENT NUMBER: 37:19038
ORIGINAL REFERENCE NO.: 37:3067c-i
TITLE: Regularities in the hydrogenative fission of N-benzyl compounds
AUTHOR(S): Birkofer, Leonhard
SOURCE: Ber. (1942), 75B, 429-41
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
OTHER SOURCE(S): CASREACT 37:19038

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AB With the higher 1-chloroparaffins, which can now be obtained independently of the alcs. by chlorination of the corresponding hydrocarbons under suitable conditions, the reaction with ammonia is considerably simpler than in the case of the lower alkyl halides. (1) Compds. higher than BuCl no longer give any appreciable amount of quaternary salt. (2) Under definite conditions of concentration, solvent, temperature and pressure, the secondary amine can be made the chief product. (3) The primary, secondary and tertiary amines differ so widely in b. p. that they can be separated by fractionation without excessive losses. A smooth formation of primary amine is apparently not yet possible. Earlier workers have not had much success with liquid NH3, even in the presence of NaNH2 or KNH2. W. and J. find that with liquid NH3 diluted about 1:1 with alc. to form a homogeneous reaction mixture, the yield of primary amine increases with the length of the alkyl chain (octyl 11, dodecyl 16, cetyl 24%). Conversely, the yield of tertiary amine decreases (trioctyl. 22, tricetyl about 0%). Under the above conditions the secondary amines are formed most easily (didodcyl 80-5%). With methylamine, the higher 1-chloroparaffins generally give the methylalkylamine along with the methyldialkylamine and-from hexyl chloride up-no quaternary salt. With the higher alkylamines (dodecyl), the secondary amine is obtained exclusively. With alkylamines above C8, practically no tertiary amine is formed. The reaction of the higher 1-chloroparaffins with secondary amines to form tertiary bases (dimethyl-, diethyl-, dibenzylalkylamines) is especially smooth; only in exceptional cases (e. g., with dicyclohexylamine) are the yields small. Of the solvents tested, MeOH and EtOH again proved suitable, but in benzene and benzine the yields were smaller than those obtained by heating the components without a solvent. The addition of tertiary amines to the higher 1-chloroparaffins to form quaternary salts could be effected, if at all, only in suitable solvents and within relatively narrow temperature ranges. Along with NMe3, dimethylalkyl- and arylamines (Me3NEt, Me3NCH2Ph, Me2NPh, etc.) are adapted to the reaction, while NEt3, NBu3, etc., react only very sluggishly. In alc. (but not in water, benzine, acetone, or without solvent) below 110° practically quant. yields of quaternary salt were obtained from octyl, dodecyl and cetyl chlorides with Me2NCH2Ph and NMe3. Above 110° the yields decrease rapidly, and at 170° no quaternary salt is obtained; the products are then chiefly the HCl salts of the tertiary bases used and long-chained tertiary amines; e. g., C12H25Cl and NMe3 at 180° give chiefly C12H25NMe2 and MeCl (resulting from the thermal decomposition of NMMe3Cl). To prepare the quaternary salts, mol. amts. of the chloroparaffin and tertiary amine can be used, but as the temperature must be kept below 110° and the consts. of the bimol. reaction are small (e. g., half-time value for 1 mol. C12H25Cl and 1 mol. NMe3 in 5 mols. alc. at 90°, about 5 h.), it is advisable to employ the tertiary amine in excess; after the reaction the excess is removed by distillation or with a solvent and reacted with fresh chloroparaffin.
Octyl chloride (40 g.) heated 20 h. at 140° in a sealed tube with 24 cc. each of liquid NH3 and alc. gave 11.4% pure octylamine (b12 76-8°), 40% dioctylamine (b3 142-7°, m. 35°), and 22% trioctylamine, b8 183-5.5°, nD19.5 1.450. C12H25Cl (35 g.), 8 cc. NH3 and 10 cc. alc. heated 19 h. at 170° gave 81% didodecylamine, m. 58°; 20 g. chloride, 20 cc. NH3 and 16 cc. alc. heated 23 h. at 110° yielded dodecylamine, b2 108-15° (isolated in 16% yield as the HCl salt, m. 183-6° (decomposition)), and 64% didodecylamine, b2 160-200°. From 18 g. cetyl chloride, 9 cc. NH3 and 7 cc. alc. heated 24 h. at 70° were obtained 24% cetylamine, b3 146-8°,

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m. 45° (HCl salt, m. 178°), and 11 g. dicetylamine, b3 about 220°, m. 65°. BuCl (30 g.), 10 g. MeNH2 and 6 cc. alc. heated 16 h. at 100-10° gave 6 g. BuNHMe (b750 85-110°) and 16 g. Bu2NMe (b750 159-60°, b11 53.5-4°, nD20 1.418; with 15 cc. alc., only 34% was obtained). Hexyl chloride (24 g.) and 26 cc. of 33% alc. MeNH2 after 16 h. at 100° gave 14 g. b755 80-110° (chiefly MeNHC6H13), and 9 g. MeN(C6H13)2, b755 228-30°, b12 118°, nD20 1.434. Octyl chloride (30 g.) and 28 cc. of 33% alc. MeNH2 heated 24 h. at 140° gave 24% MeNHC8H17, b3 60-5°, nD20.5 1.430, and 30% methyldioctylamine, b3 143-5°, nD20.5 1.443. From 32 g. dodecyl chloride and 40 cc. of 33% alc. MeNH2 after 12 h. at 160° were obtained 59% methyldodecylamine, b1.5 108-10° (HCl salt, m. 181-4°), and 37% methyldidodecylamine, b1.5 201°, m. 15-16°, nD22 1.453 (HCl salt, m. 138°), also obtained in 51% yield from 1 mol. each of the secondary amine and dodecyl chloride heated 16 h. in alc. at 160°. Cetyl chloride (60 g.) and 30 cc. of 33% alc. MeNH2 heated 18 h. at 140-50° gave 15% methylcetylamine, b1 147-50° (HCl salt, m. 169-70°), and 68% methyldicetylamine, b1 269-71°, m. 36-7°. From 7.5 g. octyl chloride and 5.5 g. Et2NH in 5 cc. alc. heated 12 h. at 160° was obtained 8 g. octyldiethylamine, b12 112-13°, nD21 1.432. Dodecyl chloride (30 g.), 20 g. Et2NH and 20 cc. alc. heated 18 h. at 140° yielded 86% diethyldodecylamine, b2 122-4°, nD19 1.443 (HCl salt, m. 119.5°); without alc. the yield was only 60% but if the heating was continued 62 h. the yield even without alc., was more than 90%; with benzine (b. 70-80°) only 50% was obtained after 20 h. Dodecyl chloride and (PhCH2)2NH in alc. at 150° gave 75% dibenzyldodecylamine, b2 219-20° (HCl salt, m. 101°). Dimethylcetylamine, b1 138°, nD23 1.445, was obtained in 82.5% yield from cetyl chloride and NHMe2 in alc. at 140°; HCl salt, m. 198°. Dimethylbenzyloctylammonium chloride (90% from octyl chloride, Me2NCH2Ph and alc. heated 24 h. at 105°), oil solidifying when cooled to 0°. Trimethyldodecylammonium chloride (75-80%), m. 37°. Dimethylbenzyldodecylammonium chloride (90% after 45 h., 50-60% after 15 h. at 90°, practically none at 170°), viscous oil. Trimethylcetylammonium chloride (almost quant. at 100-5°), m. about 70°. Dimethylbenzylcetylammonium chloride (70%), m. 58°.

ACCESSION NUMBER: 1941:22748 CAPLUS
DOCUMENT NUMBER: 35:22748
ORIGINAL REFERENCE NO.: 35:3599a-i,3600a-f
TITLE: Reaction of higher 1-chloroparaffins with ammonia, primary, secondary and tertiary amines
AUTHOR(S): Westphal, Otto; Jerchel, Dietrich
SOURCE: Ber. (1940), 73B, 1002-11
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

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GI For diagram(s), see printed CA Issue.
AB In general, the action of aqueous or alc. NH3 on organic halogen compds. is not well adapted to the preparation of primary amines; too much of the secondary and tertiary amines and even of the quaternary halide is formed, probably because, at the temps. required for the reaction, the velocities of the reactions of NH3, NH2R and NHR2 with RX are too nearly alike. The use of liquid NH3 should then favor the formation of the primary compds. Working with liquid NH3 is very simple. The reaction can be carried out in a large glass bomb tube, calibrated at its lower end, which, after the halide and the desired volume of liquid NH3 have been introduced with the necessary cooling, is sealed and kept at the desired temperature To avoid the danger of the not wholly harmless explosions which may occur, the reaction may also be carried out in a 500-1000 cc. pear-shaped steel vessel with a manometer screwed into the constricted end. After the reaction is over, the NH3 is allowed to evaporate off, the basic products are taken up in dilute HCl, and the primary, secondary and tertiary amines are separated in the usual way. With aliphatic halides, the yield of primary amine, already much higher with the lower members than in the reaction with aqueous or alc. NH3, increases rapidly with increasing mol. weight Thus, after standing 1 day in 2 vols. liquid NH3 at room temperature with frequent shaking, C5H11Br, C8H17Br and C12H25Br gave 10, 45 and 90%, resp., of primary, and 80, 43 and a few % of secondary base. Similarly, PhCH2Cl, α-C10H7CH2Cl and 9-chloromethylphenanthrene with 8 vols. liquid NH3 after 24 h. at room temperature gave 53, 72 and 70% primary and 39, 20 and 26% secondary amine, while with 3 vols. of 18% alc. NH3 at 100° they gave 9, 11 and 29% primary, 35, 38 and 25% secondary and 48, 47 and 43% tertiary base. Bis(α-naphthomethyl)amine, b0.3 230-5°, m. 55°; HCl salt, m. 230°; picrate, m. 206°; N-nitroso derivative, m. 132°. Tris(α-naphthomethyl)amine, m. 178°: HCl salt, m. 199°; picrate, m. 211°. 9-Aminomethylphenanthrene, b0.15 160-5°, m. 107°; HCl salt, m. 277°; picrate, m. 236°. sec-base, m. 193°; HCl salt, m. 239°; NO derivative, m. 268°. tert-base, m. 163°; HCl salt, m. 229°; picrate, orange-red, m. 190°. PhOCH2CH2Br gives 65% primary amine, b12 115°, with 1 part liquid NH3 after 40 h., and PhO(CH2)3Br gives 71% primary base, b15 126°, m. 130°. β-Chloroethylaniline, from PhNH2 and 10 mols. (CH2Br)2 heated 15 h. on the water bath, faintly acidified, freed from (CH2Br)2 with ether, made alkaline, extracted with ether and heated 14 h. with concentrated HCl at 100° b1 91-4° (yield, 5%); after 2 days with 5 parts liquid NH3 it gives 65% PhNHCH2CH2NH2, b15 142-4°, together with the sec-base, (PhNHCH2CH2)2NH, b0.1 215-25° (HCl salt, m. 233°; trinitroso derivative, m. 99°). Similarly, PhNMeCH2CH2Br gives 71% of the base PhNMeCH2CH2NH2, b0.3 100-12° (picrate, red, m. 174°; HCl salt, m. 205°; Ac derivative (I), b0.4 165°, m. 88°), and 20% of the triamine, (PhNMeCH2CH2)2NH, b0.3 200-2° (HCl salt, m. 204°); with 5 parts alc. NH3 20 h. at 100°, the yields of the 2 bases are 15 and 60%, resp. The green NO derivative of I, m. 140°, treated successively with NaHSO3 and HCl, yields 56% N-methylethylenediamine, b. 115-17°; HCl salt, m. 132°; picrate, m. 223°. PhNEtCH2CH2Br under the same conditions gives

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often obsd. From (CH2Cl)2 after 3 days were obtained, together with about 65% unchanged chloride, chiefly (CH2NH2)2 and (H2HCH2CH2)2NH; no piperazine was detected. With (CH2Br2)2 the reaction was complete in 10 h.; the yield of (CH2NH2)2 was much smaller and the mixt. of bases which b. up to above 250° contained a series of homologs, H2NCH2CH2(NHCH2CH2)nNH2. With very reactive halogen atoms the formation of NH at the expense of NH2 compd. may be greatly favored even with liq. NH3. Thus, (p-BrCH2C6H4)2, m. 170°, obtained in 50% yield from Ph2, 2.5 mols. HCHO and concd. HBr treated 20 h. at 50° with HBr gas, reacts rapidly with liq. NH3, yielding only about 26% of the diamine, (HNCH2C6H4)2, m. 135° (picrate, m. 222°; di-Ac deriv., m. 272°; di-Bz deriv., m. 243°); the rest of the product is a mixt. of primary-secondary bases. With alc. NH3 at 100° the yield of primary diamine is only 5%.

ACCESSION NUMBER: 1937:35287 CAPLUS
DOCUMENT NUMBER: 31:35287
ORIGINAL REFERENCE NO.: 31:4961i,4962a-i,4963a-i
TITLE: Action of liquid ammonia on organic halogen compounds
AUTHOR(S): v. Braun, Julius; Lotz, Rudolf; Warne, Kenneth C.; Pinkernelle, Walter; Rohland, Werner; Pohl, Anneliese; Dengel, Friedrich; Arnold, Herbert
SOURCE: Ber. (1937), 70B, 979-93
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

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even somewhat more of the primary base PhNEtCH2CH2NH2, b20 148-50° (HCl salt, m. 153°; picrate, m. 166°; Ac deriv., b0.5 180-5°, m. 100°), and less of the triamine, yellowish, b12 223-30° (HCl salt, m. 203°; picrate, m. 176°). PhNMe(CH2)3Cl with liq. NH3 gives 65% of the primary base, b0.3 112-15° (HCl salt, m. 189°; Picrate, red, m. 152°; Ac deriv., b0.2 168-72°, forms an olive-green NO deriv., m. 114°), and 20% of the triamine, light yellow, b0.3 220-2° (HCl salt, hygroscopic; picrate, m. 166°; Ac deriv., b0.2 250-5°, forms a light green dinitroso deriv., m. 161°). With alc. NH3 the yields of the 2 bases are 18 and 70%, resp. The above NO derivs. smoothly undergo the NaHSO3 degrdn., giving, resp., N-methyltrimethylenediamine, b. 138-9°, fumes in the air (HCl salt, m. 185°; picrate, m. 227°), and bis(γ-methylaminopropyl)amine, b15 122°, m. 22° (HCl salt, m. 275°; picrate, m. 175°). BzNH(CH2)4Cl and BzNH(CH2)5Cl with 2 parts liq. NH3 after 100 h. give 70% benzoylputrescine, b0.2 186°, and benzoylcadaverine, b0.5 202°, together with the sec bases [BzNH(CH2)4]2NH, b0.3 260°, m. 87° (HCl salt, m. 230°), and [BzNH(CH2)5]2NH, m. 69° (HCl salt, m. 199°). The compd. III (IV, R = EtO, R' = p-EtOC6H4NH), m. 118-20°, is obtained as the HCl salt, m. 231°, in 70% yield from ClCH2CONHC6H4OEt with 1 mol. PCl5 and a little POCl3; picrate, m. 118-20°. The compd. V (IV, R = H, R' = Cl) allowed to stand 2 days under liq. NH3 gives 72% of the primary base (IV, R = H, R' = NH2), m. 104-6°, becomes blue in the air when moist (HCl salt, m. 239°; picrate, m. 185°; Ac deriv., m. 170°), and 22% of the sec-base, decomps. 162-5°, yellowish when freshly pptd., becomes green, then deep blue, on standing (HCl salt, faintly greenish, m. 218-20°); alc. NH3 (18%) at 100° gives only the sec-base (90%). The anilino compd. (IV, R = H, R' = NHPh) with liq. NH3 gives 78% of the primary base, faintly yellow, m. 155° (HCl salt, m. 214°; picrate, m. 170°: Ac deriv., m. 189°, seps. with 1 H2O and is unusually hygroscopic when dehydrated), and 20% of the sec-base, m. 232° (HCl salt, yellow flocks, m. 225-30°; NO deriv., m. 119°); with alc. NH3 are obtained 38 and 50% of the primary and secondary bases. III with liq. NH3 gives 65% primary base, m. 110-12° (HCl salt, yellow, m. 143°), and 28% sec-base, m. 214-16° (HCl salt, m. 206°; Ac deriv., m. 160-2°); with alc. NH3 the yields of primary and secondary base are 30 and 50%. Cl(CH2)11Cl allowed to stand 1 day at room temp. with liq. NH3 reacts to the extent of only about 50% but what dil. HCl exts. from the product is the pure diprimary diamine, b12 140-50°. When the halogen atoms are closer together, the tendency to ring formation comes to the fore; Br(CH2)5Br and Br(CH2)4Br yield chiefly the quaternary spiranes, bispiperidinium and bispyrrolidinium bromide, and only very little cadaverine and putrescine, and also very little piperidine and pyrrolidine. The spiranes are readily isolated, by treatment with alkali, in the form of the unsatd. tertiary bases C5H10NCH2CH2CH:CH2, b. 196°, and N-butenylpyrrolidine, b. 152-4° (picrate, m. 107°; methiodide, hygroscopic, m. 178°). With Br(CH2)3Br, the tendency to ring formation disappears to a great extent and with liq. NH3 there are obtained 45-50% pure H2N(CH2)3NH2, b. 136-8° (which is thus made readily available), and 25% of the secondary-primary triamine, [H2N(CH2)3]2NH, b. 210-30°. The reaction with (CH2Cl)2 can conveniently be carried out in glass tubes, but with (CH2Br)2, when the reaction mixt. is allowed to warm up to room temp. there may occur a spontaneous evolution of heat resulting in violent explosions; in the metal bomb an increase in pressure up to 15 atm. was

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AB The reaction between an arsenous halide and an amine takes place according to the following equations: AsX3 + RNH2 → X2AsNHR.HX; X2AsNHR.HX + RNH2 → X2AsNHR + RNH2.HX; AsX3 + 2RNH2 → XAs(NHR.HX)2; XAs(NHR.HX)2 + 2RNH2 → XAs(NHR)2 + 2RNH2.HX; AsX3 + 3RNH2 → As(NHR.HX)3. The course of the reaction is influenced by several different factors, including the order of mixing, strength of the base and of the AsCl3 used. Compds. of the type XAs(NHR.HX)2 and As(NHR.HX)3 are high-melting solids, soluble in H2O (usually with decomposition), insol. in organic solvents; they resemble the corresponding NH4 halides in properties and are best regarded as As-substituted NH4 halides. Compds. of the type X2AsNHR are high-boiling liquids or low-melting solids, obtained by distillation of the solvent after removal of the precipitated NH4 halide and the insol. As compds.; they fume in the air and are decomposed violently by H2O. The name arsenamide is suggested for compds. containing the As-N linkage. In the following expts. n-C7H16 was used as a solvent. PhNH2 added to AsCl3 gave an 84.74% yield of anilinearsentriamide-3HCl, As(NHPh.HCl)3, yellow solid, decomposed by H2O, insol. in organic solvents; when the order of mixing was reversed the precipitate consisted largely of PhNH2.HCl, and on evaporation of C7H16 the filtrate yielded anilinedichloroarsenamide, Cl2AsNHPh, yellow crystalline solid, m. 89°, decomposed violently by H2O. Addition of AsCl3 to piperidine yielded 20.95% of piperidinearsentriamide-3HCl, As(NC6H10.HCl)3, long needles, m. 240-2°, decomposed by hot H2O and boiling alc.; with AgNO3 it gives the theoretical amount of piperidinearsentriamide trinitrate, m. 144°; the filtrate gave a yellow oil, b1 98°, which is probably piperidinedichloroarsenamide, Cl2AsNC5H10. Addition of AsCl3 to Et2NH gave a precipitate consisting largely of Et2NH.HCl from which no As compound could be separated; the filtrate gave diethylaminedichloroarsenamide, Cl2AsNEt2, yellow, liquid, b38 107°, fumes in the air, decomposed violently by H2O. Addition of AsCl3 to C2H4(NH2)2 gave a white precipitate from which extraction with boiling anhydrous Me2CO furnished ethylenediaminechloroarsendiamide-2HCl, ClAs(NHCH2CH2NH2.HCl)2, white solid, chars without melting above 225°; the C7H16 filtrate was not examined Addition of AsCl3 to PhNHMe gave a precipitate consisting largely of PhNHMe.HCl, from which no organic As compound could be isolated; the filtrate gave methylanilinedichloroarsenamide, Cl2AsN(Me)Ph, b3 116°, fumes in the air, decomposed by H2O. Addition of AsCl3 to benzylamine gave a precipitate from which benzylaminearsentriamide-3HCl, As(NHCH2Ph.HCl)3, was separated by sublimation at 170-200° and 2 mm. pressure, white solid, m. 246° (decomposition), decomposed by H2O and EtOH. Dibenzyl aminearsentriamide-3HCl, As[N(CH2Ph)2.HCl]3, white solid, m. 252-4° (decomposition), decomposed by H2O and EtOH, was prepared similarly from AsCl3 and dibenzylamine. Tribenzylaminearsentriamide trichloride, As[N(CH2Ph)3Cl]3, white solid, m. 209-11° (decomposition), was obtained similarly from AsCl3 and tribenzylamine. EtAsCl2 and piperidine gave a white precipitate consisting partially of piperidine-HCl, from which was separated by sublimation at 95-105° and 1 mm. pressure piperidineethylarsendiamide-2HCl, EtAs(NC5H10.HCl)2, white solid, m. 196°, decomposed by H2O; the C7H16 filtrate gave

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piperidineethylchloroarsenamide, EtAsClNC5H10, yellow, liquid, b8 108°, reacts violently with H2O to give EtAsO and piperidine-HCl. EtAsI2 and PhNH2 gave a white ppt. consisting largely of PhNH2.HI from which no As compd. could be isolated; the filtrate gave anilineethyliodoarsenamide, EtAsINHPh, light yellow oil, b10 110°, crystallizes to a yellow solid on standing, fumes in the air, reacts violently with H2O. Me2AsCl and piperidine gave a white ppt. consisting almost entirely of piperidine-HCl; the filtrate gave piperidinedimethylarsenamide, Me2AsNC5H10, colorless liquid, b8 75°, considerably more stable toward H2O than the corresponding haloarsenamides.

ACCESSION NUMBER: 1935:50647 CAPLUS
DOCUMENT NUMBER: 29:50647
ORIGINAL REFERENCE NO.: 29:6583a-i,6584a
TITLE: The arsenamides. Compounds containing the As-N linkage
AUTHOR(S): Doak, G. O.
SOURCE: Journal of the American Pharmaceutical Association (1912-1977) (1935), 24, 453-7
CODEN: JPHAA3; ISSN: 0003-0465
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

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AB cf. C. A. 28, 3051.5. NHBr2 was prepared by passing dry NH3 into Br in cold ether (3 NH3 + 2 Br2 → NHBr2 + 2 NH4Br,. A study of the decomposition rates of the NHBr2 solution at 0° and -72° shows that the product decomposes very rapidly at 0°, but it is relatively stable at the lower temperature NHBr2 reacts with RMgX to produce RNH2, R2NH, NH3 and N2. The percentage yields of these products obtained in 2 typical reactions were as follows: for BuMgCl: BuNH2 7.8%, Bu2NH 2.2%, NH3 79.0%, N2 5-9%; for PhCH2MgCl: benzylamine 29.6%, dibenzylamine 5.5%, NH3 42.8%, N2 4.7%.

ACCESSION NUMBER: 1935:19693 CAPLUS
DOCUMENT NUMBER: 29:19693
ORIGINAL REFERENCE NO.: 29:2508d-f
TITLE: The preparation of dibromoamine and its reaction with Grignard reagents
AUTHOR(S): Coleman, Geo. H.; Yager, Charles B.; Soroos, Harold
SOURCE: Proceedings of the Iowa Academy of Science (1933), 40, 112
CODEN: PIAIA9; ISSN: 0085-2236
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L12 ANSWER 240 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN
GI For diagram(s), see printed CA Issue.
AB cf. C. A. 27, 5329. An improved method is given for the preparation of the "red labile isomer" (I) (C. A. 26, 5951), the yield being 8 g. from 25 g. (.tplbond. CO2Me)2 and 10 cc. C5H5N. Heated with 50% KOH I gives 12% C5H5N, (CO2H)2, aconitic acid and a mixture of 2 compds., separated by MeCN into a dicarboxylic acid, m. 229° (also obtained from the alkaline saponification of the "yellow isomer" (II)), and a compound, C12H9O6N, analyzed as the HCl salt, m. 185° (decomposition). With 30% HBr I gives crotonaldehyde; with Hg(OAc)2 in AcOH, "Kashimoto's compound" (C. A. 27, 5329) is formed. I and 50% HClO4, heated until solution results, give the perchlorate, m. 200°, obtained also from the tribromide of II. I and (NCO2Et)2 in C6H6 give the addition complex, C23H47O12N8, m. 170°; on catalytic reduction, this takes up 8 atoms H2, giving a yellow ester; I and (NCO2Et)2 in MeOH give the previously described MeO compound, m. 160°. I with 3 mols. CH2N2 gives 2 isomeric compds. (III and IV), yellow, m. 159° (decomposition), and m. 169° (decomposition) (formulas may be interchanged). Heating the isomer, m. 159° with concentrated HCl for a short time gives a mono-Me ester of V, m. 240° (decomposition); longer heating gives pyrazoledicarboxylic acid (V), m. 260° (decomposition). Catalytic reduction of the isomer m. 159° gives the compound C18H23O8N3, m. 189°; concentrated HCl gives V. The isomer m. 169° on reduction gives the compound C18H21O8N3, m. 155°. The relation of these facts to the structure of I are fully discussed. Quinoline and (.tplbond. CCO2Me)2 in C6H6 give a "labile" addition product (VI), bright yellow, m. 177°; this is changed into the stable red isomer (VII) by heating at 195° or by the action of concentrated H2SO4 for 5 min. or concentrated HBr for several hrs. Oxidation of VI or VII with dilute HNO3 or CrO3 gives VIII, pale yellow, m. 129°. Boiling VIII with 50% KOH for 1 hr. gives the compound C14H9O4N, m. 259° (decomposition). VI (2 g.), boiled with 5 g. KOH in 300 cc. H2O, gives quinoline and (CO2H)2; with 6 g. KOH in 25 cc. H2O, 4 g. VII gives quinaldine; 5% KOH gives a dicarboxylic acid, C15H11O4N, m. 247°. VI and concentrated HCl, warmed 5 hrs., give quinoline, while VII gives the HCl salt of an acid, m. 259° (decomposition). VI and CH2N2 in C6H6 give a yellow compound (IX), C22H21O8N3, m. 153°; VII does not react with CH2N2. IX with HCl gives quinoline and V. VII is not catalytically reduced with Pd or Pt, while VI yields with Pd a dihydro derivative, yellow, m. 151°; this is unchanged after boiling 5 hrs. with concentrated HCl or concentrated KOH; oxidation gives VIII. With Pt VI gives a tetrahydro derivative, m. 177°. VI and (:NCO2Et)2 in MeOH give a MeO compound, C22H21O9N, brick-red, m. 150°; oxidation gives quinaldic acid N-oxide, m. 171° (decomposition). The stable addition product (X) of quinaldine and (.tplbond. CCO2Me)2 in AcOH, CHCl3 or MeOH gives a tetrabromide (XI), yellow, m. 145-7° (decomposition); Zn dust in boiling H2O gives X; HClO4 gives the bromoperchlorate, C22H21O8NBr.ClO4, m. 217° (decomposition). Boiling XI with HCO2H gives a dibromide, m. 145° which yields X with PhNH2. Catalytic reduction of X gives a dihydro derivative, C22H23O8N (XII), yellow, m. 164°; the labile isomer (XIII), m. 175°, gives a tetrahydro derivative, C22H25O8N, m. 175°, and also a dihydro derivative, m. 125°. Oxidation of X with HNO3 or CrO3 gives the compound C22H21O9N, pale yellow, m. 138°; catalytic reduction of this gives the compound C22H31O9N, m. 181°. XII and dilute MeOH-KOH give a compound C21H23O8N or C21H21O8N, pale yellow, m. 247-8°. X, heated with concentrated HCl for 15 hrs. at 110-20°

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and again for 16 hrs. at 225°, gives a tricarboxylic acid, C17H13O6N.H2O, decompg. 245°; distn. with CaO gives quinaldine. XIII and CH2N2 in C6H6 give the compd. C25H23O8N3, citron-yellow, m. 138°; concd. HCl gives V. The original should be consulted for the discussion of the constitution of these compds.

ACCESSION NUMBER: 1934:44979 CAPLUS
DOCUMENT NUMBER: 28:44979
ORIGINAL REFERENCE NO.: 28:5451f-i,5452a-i,5453a
TITLE: Syntheses in the hydroaromatic series. XIX. "Diene syntheses" of nitrogen-containing hetero rings. 7. The primary products in the diene syntheses of pyridine, quinoline and quinaldine
AUTHOR(S): Diels, Otto; Alder, Kurt; Friedrichsen, W.; Petersen, Ernst; Brodersen, K.; Kech, H.
SOURCE: Ann. (1934), 510, 87-128
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

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AB The products of pyrolysis of benzaldazine (I), anisaldazine, di-o-chlorobenzaldazine, p-tolualdazine (II), hydroanisamide, tri-o-chlorohydrobenzamide (III) and benzoin hydrazone (IV) are given. Lophine (V) or its corresponding derivative is obtained from I, II, III and IV. V is probably derived from I via benzalimine, the intermediate existence of which is supported by the fact that benzalfluorenoneazine on pyrolysis gives 9-iminofluorene. Benzylamine or dibenzylamine on heating yields V and tetraphenylpyrrole (VI); in the presence of stilbene only VI is obtained. The ketazines of Ph2CO and PhCOMe and the mixed ketazine of Ph2CO and fluorenone are more stable to heat than the above aldazines and tend to eliminate PhCN rather than N. The pyrolysis of I is little affected by 1000 atms. of H or N; with NH3 the reaction is complex.

ACCESSION NUMBER: 1932:54085 CAPLUS
DOCUMENT NUMBER: 26:54085
ORIGINAL REFERENCE NO.: 26:5562c-e
TITLE: The thermal decomposition of azines. A note on the thermal decomposition of benzaldazine under 1000 atmospheres pressure of nitrogen, hydrogen and ammonia
AUTHOR(S): Howard, Louis B.; Hilbert, Guido E.; Wiebe, R.; Gaddy, V. L.
SOURCE: Journal of the American Chemical Society (1932), 54, 3628-41
CODEN: JACSAT; ISSN: 0002-7863
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

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AB cf. C. A. 14, 3418. As shown in the earlier paper, tetraacetylsalicin (A), in which only the HO groups of the glucose residue are acetylated, exchanges the HO group in the side chain of the saligenin residue for Br when treated with HBr-AcOH, giving a Br derivative (B), Ac4C6H7O5OC6H4CH2Br, which served as the mother substance for the preparation of a number of compds.
described in the present paper. With Ag2CO3 it gives a product from which A was separated only after repeated crystns.; as A is otherwise easily purified, the crude product must have contained another substance, perhaps an ether-like compound which, theoretically, might be formed from 2 salicin residues in anhydrous solvents but which it has thus far not been possible to isolate. With AgNO3 B gives well crystallized compds. containing N at
first but also yielding only A after repeated purification; probably the intermediate nitrate is not stable towards alc. Better results were obtained with amines and NH3. Thus, 100 g. B under 100 cc. absolute MeOH treated with 400 cc. of an 8% solution of NH3 in MeOH and
allowed to stand 3-4 days gives 8.5 g. disalicinamine (C), NH(CH2C6H4OC6H11O5)2, needles from H2O, begins to turn yellow 200°, m. 205° (decomposition), [α]D23.5 -45.82° (N HCl), easily soluble in dilute acids; 5 g. heated 3 hrs. on the H2O bath with 50 cc. of 5%
HCl in a slow current of CO2 gives 1.13 g. (o-HOC6H4CH2)2NH, needles from alc., m. 168°, easily soluble in dilute acids and alkalies. The mother liquors from the C on evaporation in vacuo yield trisalicinamine as an oil which, heated 1 hr. on the H2O-bath with 300 cc. Ac2O and 50 g. NaOAc, poured with stirring into 2 l. cold H2O, neutralized with NaHCO3 after several hrs., filtered, rubbed with 100 cc. warm MeOH to remove impurities and crystallized from 10 parts alc. gives 27 g. of dodecaacetyltrisalicinamine,
microneedles, m. 173-5°, [α]D24 -45.13° (CHCl3), easily soluble in dilute acids; 10 g. heated 3 hrs. on the H2O-bath in CO2 with
5% HCl gives 1.8 g. tri-[o-hydroxybenzyl]-amine hydrochloride, stout needles, begins to decompose 110°, difficultly soluble in cold, easily in hot dilute acids and in dilute alkalies. Pentaacetylsalicinmethylamine (D), obtained in 20.3% yield from B and MeNH2 in MeOH shaken 2 hrs., allowed to stand 12 hrs., evaporated in vacuo to a sirup and heated 1 hr. on the H2O bath with Ac2O-NaOAc, stout tablets from 50% MeOH, m. 165°. [α]D29 -37.68° (CHCl3), hydrolyzed by 5% HCl to o-hydroxybenzylmethylamine, precipitated as the phosphotungstate and isolated as
the hydrochloride (yield, 44.6%), fine needles from MeOH-Et2O, m. 130°. The AcOH mother liquors from D, neutralized with solid NaHCO3, give 60 g. crude and 31 g. pure [octaacetyldisalicin]methylamine, needles from Me2CO, m. 198-200°, [α]D24 -35.40° (CHCl3). Pentaacetylsalicinethylamine, prepared like D (yield, 13.8%), needles from 50% alc., m. 96-7°; [octaacetyldisalicin]ethylamine (yield, 20%), long needles from alc., m. 151-3°. Salicindiethylamine, from B and NHEt2 (yield, 63.5%), needles from petr. ether, m. 102-3°, [α]D30 -26.05° (CHCl3), has a very bitter taste. [Tetraacetylsalicin]-methylphenylamine (tetraacetylsalicin-N-methylaniline), from B and PhNHMe in MeOH (yield, 76%), long needles from MeOH, m. 140-1°, [α]D30 -19.86° (CHCl3), gives in MeOH on the H2O bath with NH4OH 70.2% salicinmethylphenylamine, [α]D30.5 -36.23° (Me2CO). Tetraacetylsalicintrimethylammonium bromide, from B and NMe3 in alc. (yield, 91.5%), needles, sinters 65°, m. 68°, [α]D26

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-42.37° (H2O), tastes very bitter, hydrolyzed by HCl to o-hydroxytrimethylammonium chloride (purified through the phosphotungstate and obtained in 66% yield), fine needles with 1 H2O from MeOH-Et2O, m. 96° (anhyd., 200° (decompn.)).

ACCESSION NUMBER: 1922:21356 CAPLUS
DOCUMENT NUMBER: 16:21356
ORIGINAL REFERENCE NO.: 16:3651h-i,3652a-g
TITLE: New nitrogen-containing derivatives of salicin and polynuclear hydroxybenzylamines
AUTHOR(S): Zemplen, Geza; Kunz, Alphons
SOURCE: Ber. (1922), 55B, 979-92
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

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GI For diagram(s), see printed CA Issue.
AB S. has found that tetranitromethane (a) is decomposed by aqueous alkalies in 2
ways: (1) a + 2KOH = KNO3 + KC(NO2)3 + H2O (Hantzsch and Rinkenberger, Ber. 32, 629(1899)), and (2) 3a + 6KOH = 4KNO2 + K2CO3 + 3H2O. The relative extent of each reaction depends on the concentration of the alkali, (1) increasing from 66-47% with 0.1 N KOH to 92.30% with 14.04 N KOH. Iodotrinitromethane (b), which with AgNO2 almost instantly gives a, is decomposed by alkalies only according to the equation 3b + 6KOH = 3KC(NO2)3 + 2KI + KIO3 + 3H2O (Hantzsch, Ber. 39, 2479(1906)). Reaction (1) led Willstatter and Hottenroth to conclude that in a two of the NO2 groups have a peculiar position and they assigned the structure (OZN)2-C.O.NONO2 to a (Ber. 37, 1797(1904)), and since b gives only CH(NO2)3, S. believes that reaction (2) depends on the fourth "nitro" group; the formation of KNO2 makes the presence of a .tplbond. CONO grouping in a probable, as in the structure (OZN)3CONO; both forms of a are in equilibrium, the first being the more stable in concentrated alkalies. In analyzing the decomposition products, the HNO2: was determined by Gerlinger's method (boiling with NH4Cl and determining as N (Z. angew. Chemical 1901, 1250); by using Ba(OH)2 instead of KOH for the decomposition, the CO2 could be determined as BaCO3; the HNO3 was determined by means of nitron after the CH(NO2)3: present bad been converted by H and Pd into a substance of as yet unknown constitution which dogs not react with nitron (the reduction of HNO3 to HNO3 under these conditions is negligible); the CH(NO2)3 can be determined by distilling the solution, after boiling off the HNO2: with NH4Cl and adding a few cc. of 84% H3PO4, and determining it in the distillate with nitron (very little HNO3 distils over). The Pd catalyst used in the reduction of the CH(NO2)3 is prepared by treating 20 parts BaSO4 (precipitated hot) suspended in 400 parts hot H2O with 1.7 parts PdCl2: in 50 parts H2O and 1 part of 40-50% HCHO, making faintly alkaline to litmus with NaOH, boiling until the liquid is clear and colorless, filtering, washing the gray precipitate with hot H2O to neutral reaction, drying in vacua over KOH and powdering. In acid medium, also, a decomps. into HNO2; thus 5 g. m-MeC6H4NMe2 in 20 cc. alc. and 3.1 cc. HCl (d. 1.19) heated on the H2O bath with 2.4 g. a gives 54% Me(ON)C6H4NMe2 For the quant. estimation of CH(NO2)3 in its compds., about 0.12 g. of the substance in 100 cc. H2O on the H2O bath, acidified with 1 cc. AcOH, is treated with 10-12 cc. of 10% nitron acetate and after standing 2 hrs. in ice the precipitate is filtered on a Gooch crucible, washed with 5 cc. ice H2O in small portions and dried in vacuo over P2O5; the nitron nitroform, CH(NO2)3C20H16N4, decomps. 136-41°. The following nitroform salts were prepared by neutralizing aqueous solns. of CH(NO2)3 with the corresponding base: Di-isobutylamine, (C4H9)NH.CH(NO2)2, felted needles from EtOH-H2O (1:2), decomps. 121-3°, decomps. on standing; piperidine, serrated leaves from AcOEt-CHCl3 (1:2), decomps. 100°, begins to liquefy after a time; dibenzylamine, needles from EtOH-H2O (3:5), decomps. 160-3°. That the failure to detect HNO2 in the decomposition products of b was not due to its conversion into HNO3 by the I in the alkaline solution was shown experimentally; I does not react with nitrites in alkaline solution

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ACCESSION NUMBER: 1919:12061 CAPLUS
DOCUMENT NUMBER: 13:12061
ORIGINAL REFERENCE NO.: 13:2362d-i,2363a-b
TITLE: Constitution of tetranitromethane
AUTHOR(S): Schmidt, Erich
CORPORATE SOURCE: Berlin
SOURCE: Ber. (1919), 52B, 400-13
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
OTHER SOURCE(S): CASREACT 13:12061

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              structure diagram, plus NTE and SEQ fields
FHITSTR ----- First HIT RN, its text modification, its CA index name, and
              its structure diagram
FHITSEQ ----- First HIT RN, its text modification, its CA index name, its
              structure diagram, plus NTE and SEQ fields
KWIC -------- Hit term plus 20 words on either side
OCC --------- Number of occurrence of hit term and field in which it occurs

To display a particular field or fields, enter the display field
codes.  For a list of the display field codes, enter HELP DFIELDS at
an arrow prompt (=>).  Examples of formats include: TI; TI,AU; BIB,ST;
TI,IND; TI,SO.  You may specify the format fields in any order and the
information will be displayed in the same order as the format
specification.
```

All of the formats (except for SAM, SCAN, HIT, HITIND, HITRN, HITSTR, FHITSTR, HITSEQ, FHITSEQ, KWIC, and OCC) may be used with DISPLAY ACC to view a specified Accession Number.
ENTER DISPLAY FORMAT (BIB):occ

L12 ANSWER 1 OF 243 CAPLUS COPYRIGHT 2005 ACS on STN

FIELD	COUNT
AB	1
ST	1
IT	10

```
=> s dibenzylamine
          2667 DIBENZYLAMINE
            38 DIBENZYLAMINES
L13       2688 DIBENZYLAMINE
                (DIBENZYLAMINE OR DIBENZYLAMINES)

=> d his

     (FILE 'HOME' ENTERED AT 16:14:00 ON 11 APR 2005)

     FILE 'REGISTRY' ENTERED AT 16:14:08 ON 11 APR 2005
L1             1 S DIBENZYLAMINE/CN

     FILE 'CAPLUS' ENTERED AT 16:14:43 ON 11 APR 2005
L2          1990 S 103-49-1/RN
L3        408778 S ?COLOR
L4          1791 S ?COLOUR
L5        409531 S L3 OR L4
L6            28 S L2 AND L5
L7       1658396 S PUR?
L8       1454481 S STAB?
L9            131 S L2 AND L7
L10           138 S L2 AND L8
L11           256 S L9 OR L10
L12           243 S L11 NOT L6
L13          2688 S DIBENZYLAMINE

=> s l13 and l5
L14           72 L13 AND L5

=> d l14 not l6
L6 IS NOT VALID HERE
For an explanation, enter "HELP DISPLAY".

=> s l14 not l6
L15           49 L14 NOT L6

=> s l15 not l11
L16           49 L15 NOT L11

=> s l16 not l12
L17           49 L16 NOT L12

=> d l17 1-49 abs ibib
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L17 ANSWER 1 OF 49 CAPLUS COPYRIGHT 2005 ACS on STN
AB Fifteen diamagnetic CuBrL complexes of Cu(I) were prepared in anhydrous EtOAc
by adding an EtOAc solution containing an excess of ligand (L = amine or heterocyclic base) to a solution of CuBr (ligand, color, m.p. given): PhCH:NPh, black, 156°; Ph2NH, green, 243° (decompose); PhNHEt, black, 218° (decompose); Me2NCH2CH2CH2NH2, green, 218° (decompose); C6H4NHPh, black, 191°; PhCH2NHPh, black, 251°; (PhCH2)2NH, green, 106°; PhNHMe, black, 127°; PhNEt2, dark brown, 120°; PhNMe2, steel gray, 117°; α-picoline, brown, 224°; β-picoline, green, 206°; γ-picoline, yellow-brown, 165°; piperidine, light brown, 211° (decomposition); piperazine, green, 140° (decompose). The compds. were semicryst. powders, stable in dry air at room temperature, and insol. in nonpolar solvents. They dissolved in dilute acids. The ir spectra were recorded for the CuBr complexes with Ph:NPh, Ph2NH, γ-picoline, and piperazine. The free amine band at .apprx.3470 cm.-1 was shifted to 3000-460 cm.-1 in the complexes. No structural change in the C6H6 ring or C-N band on coordination was evident.
ACCESSION NUMBER: 1968:92574 CAPLUS
DOCUMENT NUMBER: 68:92574
TITLE: Complexes of cuprous bromide with secondary and tertiary amines and heterocyclic bases in nonaqueous media
AUTHOR(S): Prasad, Sarju; Trivedi, S. R. C.
CORPORATE SOURCE: Banaras Hindu Univ., Varanasi, India
SOURCE: Journal of the Institution of Chemists (India) (1968), 40(Pt. 1), 9-14
CODEN: JOICA7; ISSN: 0020-3254
DOCUMENT TYPE: Journal
LANGUAGE: English

L17 ANSWER 2 OF 49 CAPLUS COPYRIGHT 2005 ACS on STN
AB Urbach's light sensitive system was used to test the effectiveness of sun screening agents. Red Veterinary Petrolatum, Red Veterinary Petrolatum with vitamin B2, 2-ethylhexyl salicylate, 2-ethoxyethyl p-methoxycinnamate, homomenthyl salicylate, iso-Bu p-aminobenzoate, p-aminobenzoic acid, and 2-hydroxy-4-methoxybenzophenone-5-sulfonic acid were tested. The Urbach system consists of a mixture of 62 mg. methyl yellow, 120 mg. hexachlorocyclopentadiene, 10 mg. dibenzylamine, and 447 g. Paraplast. The wax is melted and the other materials are added. The melt is poured in uniform layers into Petri dishes. A brass plate 167 μ thick, which fits inside the Petri dish, is pierced with a center hole and 8 holes equally spaced around the center hole. Each hole is 6 mm. in diameter The material under test was mixed with melted polyethylene glycol 1500 except in case of Red Veterinary Petrolatum and a mixture of this with vitamin B2. Fifty mg. of one of these mixts. was placed in each of the peripheral holes and plain propylene glycol 1500 in the center hole, and smoothed off to form an even layer. The dish was then exposed to a Westing-house S.S. 20 fluorescent sunlamp at a distance of 25.5 cm. for 20 min. On the basis of the change in color of the Urbach wax, the sunscreen agents were classified as good, fair, and poor. The results obtained do not confirm results obtained by the spectral absorption method but are more nearly in line with results actually obtained in use on the skin. However, for absolute certainty, actual testing on a fairly large number of human subjects may be required.
ACCESSION NUMBER: 1967:108175 CAPLUS
DOCUMENT NUMBER: 66:108175
TITLE: Evaluation of sunscreen agents
AUTHOR(S): Das Gupta, Vishnu
CORPORATE SOURCE: Sch. of Pharm., Univ. of Georgia, Athens, GA, USA
SOURCE: Journal of the Society of Cosmetic Chemists (1967), 18(3), 143-7
CODEN: JSCCA5; ISSN: 0037-9832
DOCUMENT TYPE: Journal
LANGUAGE: English

L17 ANSWER 3 OF 49 CAPLUS COPYRIGHT 2005 ACS on STN
GI For diagram(s), see printed CA Issue.
AB cf. CA 61, 16032c. Refluxing 90 g. 6-methylthio-3-methylbenzyl chloride with 400 g. urotropine and 430 ml. 50% AcOH 3 hrs., followed by addition of 153 ml. concentrated HCl and heating 5 min. longer, gave after extraction with C6H6 80% 6-methylthio-3-methylbenzaldehyde (I), b3 125-7°, m. 26-6.5°; 2,4-dinitrophenylhydrazone m. 253-4°. I (20 g.) in Et2O was added to liquid NH3 under argon atmospheric, followed by 6.6 g. Na added gradually to give a stable blue color; excess Na was decomposed with NH4Cl, the mixture evaporated and treated with aqueous NH4OH and C6H6 to yield 64.5% C16H15NS2, possibly 3,9-dimethyl-6,12-iminodibenzo-[b,f]-[5,11],dithiocin, (II), m.206-6.5°. Also formed was 2-hydroxymethyl-4-methylthiophenol, b5 135-8° (with some decomposition), which gave the Hg salt, m. 198-9°; disulfide m. 95-6°. In expts. in which all traces of residual NH3 were removed by heating prior to the aqueous treatment of the reaction mixture, there was also formed 6-thiolo-3-methylbenzoic acid, isolated as the corresponding disulfide, m. 290-1°. II and Ac2O gave N-acetyl derivative, m. 201-2°, which with Ranay Ni in C6H6 9 hrs. at 50-60° gave 71.5% N,N-bis(3-methylbenzyl)acetamide (III), b0.3 149-50°. III heated with aqueous HCl gave the free amine, isolated as HCl salt, m. 197.5-8°.
ACCESSION NUMBER: 1967:55447 CAPLUS
DOCUMENT NUMBER: 66:55447
TITLE: Action of sodium in liquid ammonia on 6-methylthio-3-methylbenzaldehyde
AUTHOR(S): Gol'dfarb, Ya. L.; Skorova, A. E.; Kirmalova, M. L.
CORPORATE SOURCE: N. D. Zelinskii Inst. Org. Chem., Moscow, USSR
SOURCE: Izvestiya Akademii Nauk SSSR, Seriya Khimicheskaya (1966), (8), 1421-5
CODEN: IASKA6; ISSN: 0002-3353
DOCUMENT TYPE: Journal
LANGUAGE: Russian
OTHER SOURCE(S): CASREACT 66:55447

L17 ANSWER 4 OF 49 CAPLUS COPYRIGHT 2005 ACS on STN
AB Complex compds. containing 1 mol. TiCl3, 2 mols. of a secondary or tertiary amine, and 1 mol. of EtOAc have been prepared Anhydrous TiCl3 was prepared by the reduction of TiCl4 with finely divided Al powder at 190°. The black mass of TiCl3 was extracted with anhydrous EtOAc and filtered. Complexes were prepared by addition of the amine solution in EtOAc in small quantities to TiCl3 solution in such a way that TiCl3 was in slight excess. The product was filtered in a dry atmospheric, washed with EtOAc, pressed between filter paper, and then dried in a vacuum desiccator. The compds. are colored and fairly stable. They are insol. in nonpolar organic solvents, soluble in dilute mineral acids, slightly soluble in EtOH, and hydrolyze in H2O. Some, such as the compds. formed with methylaniline, N-benzylaniline, and tribenzylamine are slightly soluble in Me2CO. All of the compds. lose weight corresponding to 1 mol. of EtOAc when heated at 100°. On further heating, some of them give a sharp m.p. while others melt with decomposition The following compds. were prepared which have the probable formula TiCl3.2A.EtOAc (A, color of complex, and m.p. given): dibenzylamine, cream yellow, 160°; dimethylaniline, light brown, 280° (decomposition); N,N'-diphenylbenzidine, cream yellow, 300° (decomposition); N-benzylaniline, cream yellow, 210° (decomposition); benzalaniline, yellow turning to apple green, 200° (decomposition); ethylaniline, dirty cream, 170°; tribenzylamine, cream yellow, 130°; Et2NH, light brown, 185° (decomposition); MeNH2, dirty green, 200°; Et3N, dirty cream, 200°; diethylaniline, light brown, 240° (decomposition); and Ph2NH, orange red turning to apple green, 255°.
ACCESSION NUMBER: 1967:16134 CAPLUS
DOCUMENT NUMBER: 66:16134
TITLE: Complex formation of anhydrous titanium(III) chloride with secondary and tertiary amines
AUTHOR(S): Prasad, Sarju; Devi, K. Shyamala
CORPORATE SOURCE: Banaras Hindu Univ., Varanasi, India
SOURCE: Journal and Proceedings of the Institution of Chemists (India) (1966), 38(4), 178-80
CODEN: JPICAE; ISSN: 0368-3648
DOCUMENT TYPE: Journal
LANGUAGE: English

L17 ANSWER 5 OF 49 CAPLUS COPYRIGHT 2005 ACS on STN

AB Twenty-eight substances known to affect the mammalian autonomic nervous system were injected into intact P. phoxinus. The responses of the melanophores were recorded and the reactions of Phoxinus and mammals were compared. The same substances were applied and the melanophore responses studied in isolated pieces of skin, in whole animals during elec. stimulation, and in animals whose spinal cords and (or) spinal nerves had been sectioned. No evidence was obtained for the presence of cholinergic pigment-dispersing fibers. Marked pigment-dispersing effects were obtained only with substances which interfere with the normal working of adrenergic mechanisms, or with transmission in sympathetic ganglia in mammals, e.g., adrenergic blocking agents, depleters of catechol amines, and ganglionic blocking agents.

ACCESSION NUMBER: 1966:501784 CAPLUS
DOCUMENT NUMBER: 65:101784
ORIGINAL REFERENCE NO.: 65:19048h,19049a
TITLE: The effects of drugs on the background color response of the minnow Phoxinus phoxinus
AUTHOR(S): Healey, E.G.; Ross, D. M.
CORPORATE SOURCE: Univ. London
SOURCE: Comparative Biochemistry and Physiology (1966), 19(3), 545-80
CODEN: CBCPAI; ISSN: 0010-406X
DOCUMENT TYPE: Journal
LANGUAGE: English

L17 ANSWER 6 OF 49 CAPLUS COPYRIGHT 2005 ACS on STN

GI For diagram(s), see printed CA Issue.

AB cf. CA 63, 2952c. p-ONC6H4NEt2 (I), p-ONC6H4N(CH2Ph)2 (II), and p-nitrosophenylmorpholine (III) were condensed with 1,2-dimethylquinolinium perchlorate (IV) and the 1,4-isomer (V) of IV to give the corresponding anils and with 1-methyl-2-(pyridiniomethyl)pyridinium diperchlorate (VI) and the 1,4-isomer (VII) or VI to the corresponding nitrones. The diiodide analog of VI in a little H2O treated with excess saturated aqueous NaClO4 yielded 75% VI, m. 263-4°. Similarly was prepared VII, 85%, m. 236-7°. PhN(CH2Ph)2 (20 g.) in 300 cc. absolute EtOH and 16 g. concentrated H2SO4 treated dropwise at 5° with stirring with 13 g. iso-AmONO yielded 11 g. green II, m. 94-5° (EtOH). IV (0.01 mole) in 50 cc. hot MeOH treated with 0.01 mole appropriate nitroso compound and then 3 drops piperidine yielded the corresponding VIII (X, m.p., color, and % yield given): Et2N, 204-6° (HCONMe2-EtOH), dark green with a metallic luster, 50; (PhCH2)2N, 215-17° (HCONMe2-EtOH), brown-violet to black-green, 55; morpholino, 240-5° (HCONMe2-EtOH), black-green, 70. VII gave similarly the corresponding IX (same data given): Et2N, 216-18°, dark green with a metallic luster, 70; (PhCH2)2N, 200-2°, brown-violet to dark green, 75; morpholino, 150-5° (or 195° on slow heating), dark green, 70. VI (0.01 mole) in 20 cc. hot H2O or the VII in 30 cc. hot H2O treated with stirring with 0.01 mole appropriate nitroso derivative and 1 cc. piperidine in 20 cc. MeOH yielded the corresponding X; in the runs with II, 0.01 mole each of the reactants in 30 cc. HCONMe2 treated with 1 cc. piperidine and after a few min. diluted with 150 cc. EtOH gave the corresponding X. In this manner were prepared the following X (X, position of the side-chain, m.p., color, and % yield given): Et2N, 2, 136-8° (EtOH) (red) (or about 95° (red-brown with green luster), red, 80; (PhCH2)2N, 2, 168-71° (HCONMe2-EtOH), red-brown to brown-violet, 90; morpholino, 2, 174-6° (HCONMe2-EtOH), red-brown, 75; Et2N, 3, 180-2° (HCONMe2-EtOH), black-violet with a green luster, 90; (PhCH2)2N, 3, about 180° (HCONMe2-EtOH), red-brown, 85; morpholino, 3 (containing 1 mole HCONMe2), 105-8° and 185-90°, brown-violet, 90.

ACCESSION NUMBER: 1966:75702 CAPLUS
DOCUMENT NUMBER: 64:75702
ORIGINAL REFERENCE NO.: 64:14165a-f
TITLE: Azomethines with nitrogen mustard groups. VIII. Anils and nitrones from p-nitroso-N,N-diethylaniline, p-nitroso-N,N-dibenzylaniline, and p-nitrosophenylmorpholine as comparison substances without nitrogen and mustard groups
AUTHOR(S): Schulze, Werner; Willitzer, Horst
CORPORATE SOURCE: Deut. Akad. Wiss., Berlin
SOURCE: Journal fuer Praktische Chemie (Leipzig) (1966), 31(3-4), 131-5
CODEN: JPCEAO; ISSN: 0021-8383
DOCUMENT TYPE: Journal
LANGUAGE: German

L17 ANSWER 7 OF 49 CAPLUS COPYRIGHT 2005 ACS on STN

GI For diagram(s), see printed CA Issue.

AB A method is described for preparing colored reproductions by electrography. Permanent reproductions are obtained by selectively depositing and irreversibly bonding a H2O-insol. organic compound to the surface of a dye-sensitized photoconductor on an elec. conductive carrier by electrolytically decomposing onium ions. Iso-BuCOMe (139 g.), 252 g. ZnO (having a particle size <10 μ), 210 g. 30% 3:7 butadiene-styrene copolymer in MePh, 50 cc. 0.5% Acid Blue 1 in MeOH, 20 cc. 0.5% Acid Red 92 in MeOH, and 5 cc. Basic Red 92 in MeOH ground 20 min. in a Waring Blendor, filtered through a coarse glass filter, and coated onto an Al sheet, and the sheet dried in the dark in warm air and dark-adapted during 24 hrs. gave a photoconductive sheet with a high response at 460-5, 560, and 660 mμ. ZnO 34.4, Pliolite E-7 29.6, and Me2CO 11.8 milled 8 hrs., diluted with AcOEt 23, mixed with 0.5% Phosphine R-MeOH 2 and 0.5% Xylene Cyanol FF-MeOH 0.6 part, and coated in the usual manner gave a photoconductive layer. The photoconductive sheet placed with its Al backing onto the metal base (neg. electrode) of a developing tray, exposed to light under a negative, a pos. electrode placed in the developer tray which was then filled with the desired onium salt solution, and a 30-v. current passed 10 sec. through the photoconductor sheet which was then washed with hot H2O (about 140°F.) and dried gave a pos. color image; if reexposure of the sheet is desired, dark adaptation is again required. Alcian Blue 8G N (5 g.) in 100 cc. H2O similarly gave a cyan image. Bis(chloromethyl)-4,4'-bis(6-methyl-2-benzothiazolyl)azobenzene (5 g.) and 25 g. CS(NMe2)2 (I) pasted with H2O and heated 1 hr. at 90°C. gave a yellow thiuronium salt which yielded yellow images by the process of this invention. Coupling product (5 g.) from Naphthol AS-LG and Fast Red Salt FRN in 75 g. 100% H2SO4 treated at 0°C. with 25 g. ClCH2OMe, stirred 25 min. at 60°C., and poured onto ice, and a 5-g. portion of the product pasted with 25 g. I and a little H2O, heated 1.5 hrs. at 90°C., and diluted with 200 g. Me2CO gave a dark precipitate which, as a 5% aqueous solution, gave yellow images on the ZnO conductor sheets. Indigo Yellow (2.5 g.) and 5 g. I gave similarly a gum which in aqueous solution gave yellow images. BzCH2CO2Et condensed with 2,5-(MeO)2C6H3NH2 in boiling xylene, the product coupled in C5H5N with the diazotized amine obtained by condensing p-AcNHC6H4SO2Cl with Et2NCH2CH2NH2, and the coupling product hydrolyzed gave a yellow azo dye; a 2.9-g. portion heated 20 hrs. on the steam bath with 2 g. BzCH2Br and 0.5 g. NaHCO3 in 50 cc. 95% EtOH gave a yellow solid which produced brilliant yellow dye images with a strong metallic luster on the surface of ZnO photoconductor sheets; a 3-g. portion of the azo dye in 25 cc. AcOH stirred 1 hr. on the steam bath with 2 cc. (ClCH2)2O, and the product heated 1 hr. on the steam bath with 10 g. I and poured into 200 cc. boiling C6H6 gave a solid which produced yellow images with a bronze luster. Basolan Chrome Brilliant Red 3BM (15 g.), 40 cc. SOCl2, and 1 drop C5H5N kept overnight, the resulting chloride (11.2 g.) treated slowly with stirring with 5.5 g. p-OZNC6H4NH2 in 30 cc. dry HCONMe2 and then dropwise with 5 cc. C5H5N and heated 0.5 hr. on the steam bath, the product dissolved in 100 cc. C5H5N, treated with a few drops HCl and slowly with 15 g. powdered Fe, heated 1 hr., and diluted with H2O to 1 l., the precipitate (3 g.) treated with 10 cc. ClCH2COCl and 2 g. AcOK, and the resulting red-brown solid heated 0.5 hr. on the steam bath with 15 g. I gave a reddish gum which produced magenta images. Anthragen Red Violet RHC (5 g.) treated successively with 25 g. (ClCH2)2O and 20 g. I gave a solid which produced reddish purple images, p-AcNHC6H4NH2 treated with

L17 ANSWER 7 OF 49 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)

2,3-HOC10H6CO2H and PCl3 in hot MePh, heated several hrs. with dil. aq. KOH, and coupled with diazotized 4,2-ClMeC6H3NH2 in C5H5N-HCONMe2, and the product treated successively with ClCH2COCl and I gave a thiuronium salt which produced magenta images. C14H29NH2 (8.5 g.) and 3.9 g. KOAc in 50 cc. MeOH added during 15 min. to 10 g. p-ClCH2C6H4SO2Cl in 80 cc. MeOH and stirred 2 hrs. yielded 6 g. p-ClCH2C6H4SO2NHC14H29 (II). II (1.0 g.) and 0.4 g. I heated several min. at 110°C. gave 1.2 g. gelatinous thiuronium salt (III). III deposited a colorless neg. image on a ZnO photoconductor sheet; the areas so coated were H2O-repellent and were preferentially dyed by an aq. Basolan Chrome Brilliant Red 3BM soln.; the unexposed portions which were not coated can be removed with HCl and AcOH, or can be preferentially dyed with an acid-sol, azo dye, or can be rendered hydrophilic with aq. borax to give a sheet which can be inked for use as a lithographic plate in the hydrophobic portions. III soln. contg. a suspended pigment from Naphthol AS-LG and Fast Red Salt ITRN gave a neg. yellow image on the exposed portions of the photoconductor sheet; a black image was obtained when the soln. contained carbon black. A 0.5% aq. soln. (100 cc.) of [p-C14H29NHCOC6H4NMe3]Cl and 100 mg. 5-[2,5-MeO(Et2NSO2)C6H3N:NCHBzCONH] deriv. of 2-heptadecylbenzimidazole gave a clean bright yellow image. Dye IV (50 mg.) and 1 g. Beetle Resin 227-8 in 10 cc. EtOH added to 100 0.5% aq. [p-C14H29NHCOC6H4NMe3]Cl and used as the electrolyte deposited a purple-black image. C16H33NMe22 (13 g.) and 10 g. BzCH2Br in 60 cc. dry C6H6 kept 72 hrs. at room temp. gave [C16H33NMe2CH2CH2Bz]Br (V). V (0.6 g.), 0.5 g. zein, and Azosol Fast Yellow GT in 10 cc. hot EtOH added to 100 cc. H2O with stirring gave a dispersion which produced bright yellow images; this soln. was also used for the 3rd image to make full color images in 3 stages with the 1st image obtained with Alcian Blue 8GN and the 2nd with Astraphloxin FF. V (0.6 g.), 0.2 g. Azosol Fast Red 3 BA, and 0.2 g. zein in 10 cc. EtOH added to 100 cc. H2O gave a dispersion which produced magenta images. Cyanuric chloride condensed in Me2CO in the presence of NaHCO3 with N1-tetradecylsulfanilamide, and the product heated 0.5 hr. with 5 parts I on the steam bath and dild. with H2O gave a soln. which deposited a colorless oily neg. image, p-ClCH2C6H4COCl (18.5 g.) in 150 cc. hot xylene refluxed 1.5 hrs. with 6 g. 1,5-diaminoanthraquinone (VI), treated with an addnl. 6 g. VI and heated 3 hrs., and the resulting yellow solid, m. 224-40°C., heated on the steam bath with 10 parts I gave a thiuronium salt which produced yellow images. A ZnO sheet exposed in all areas to white light, colored by electrolytic deposition with Alcian Blue 8GN, dark adapted by contacting with hot H2O and drying, exposed to a photographic image, contacted with a suspension of TiO2 in dil. aq. III, and subjected to the electrodeposition process deposited selectively TiO2 in the light-struck areas of the sheet producing thus a pos. image formed by the remaining blue-green areas. Full color pos. images can also be obtained by this process by employing a photoconductor sheet which is colored by a mosaic pattern of yellow, cyan, and magenta sites.

ACCESSION NUMBER: 1965:424661 CAPLUS
DOCUMENT NUMBER: 63:24661
ORIGINAL REFERENCE NO.: 63:4438f-h,4439a-h,4440a
TITLE: Photoconductography employing organic onium ions
INVENTOR(S): Tulagin, Vsevolod; Coles, Robert F.; Miller, Richard A.
PATENT ASSIGNEE(S): Minnesota Mining and Manufacturing Co.
SOURCE: 7 pp.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

L17 ANSWER 7 OF 49 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 3172826		19650309	US	19600418
GB 990971			GB	

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AB cf. Bonitz, CA 50, 164f. From R2AlH and azomethines or secondary amines were prepared R2-AlNR2', which existed as associated compds. In spite of this, the compds. formed mol. compds. [electron-donor-acceptor (EDA) complexes] with strong electron donors. The EDA complexes were colored when the ligand was an azomethine or aromatic N-heterocycle. (All expts. were conducted in an argon atmospheric with exclusion of light and moisture; solvents were dried by distillation from K-Na alloy, freed of air, and withdrawn under argon; m.p. determined under argon in sealed 1-2 mm. tubes.) p-MeC6-H4N:CHPh (I), m. 44°, PhCH2N:CHPh (II), b0.001 93°, p-MeC6H4N:CHC6H4Me-p (III), β-C10H7N:CHPh (IV), m. 100-1°, α-C10H7N:CHC10H7-α (V), m. 113-15°, and PhN:CHPh (VI), m. 56°, were prepared R2AlH (VII) (R = iso-Bu throughout this abstract) (15.6 g.) in 20 cc. C6C6 treated gradually at room temperature with 18.1 g. VI in 40 cc. C6H6, and the mixture stirred several min. until the initial red color turned yellow gave 27.8 g. R2-AlNPhCH2Ph (VIII), m. 102-5°, yielding, on methanolysis in C6C6, PhNHCH2Ph (IX), b0.001 100°, m. 37°. IX and a slight excess VII in C6H6 heated until the calculated amount H was evolved gave VIII. VII (14.9 g.) in 10 cc. C6H6 was treated dropwise with 21.9 g. III in 60 cc. C6H6 with stirring and moderate cooling to give 21.7 g. R2AlN(CH2C6H4Me-p)C6H4Me-p. V and VII treated similarly gave R2AlN(CH2C10H7-α)C10H7-α (X). Phenanthridine (17.9 g.) in 40 cc. hot C6H6 treated gradually with 15.6 g. VII with stirring and external cooling, and when the exothermic reaction subsided, the mixture stirred 10 min. until it became color -less gave 28.1 g. R2AlZ (Z = 5,6-dihydrophenanthridin-5-yl) (XI) decomposed 162-5°. 9,10-Dihydrophenanthridine (XII) in C6H6 added dropwise at 0° to a slight excess of VII in C6H6 gave 91% XI, decomposed 165°. From Et2AlH and PhNHMe was prepared Et2AlNMePh, b0.005 190° (decomposition). Addition of 13.4 g. Ph-NHMe in C6H6 to 19.2 g. VII at 0° gave 21.8 g. R2AlNMePh, decomposed 110-14° (C6H6-pentane); on distillation in vacuo isobutene was partially eliminated. From Ph2NH and VII was prepared 50-60% R2AlNPh2, decomposed 80-5° (softens above 70°). Similarly, Bu2AlH and Ph2NH gave Bu2AlNPh2 (XIII). Bu3Al (30.6 g.) and 25 g. Ph2NH in C6H6 boiled 3 h. (3.4 l. pure butane was evolved) gave 27 g. XIII, m. 85-6° (slight decomposition). VI (36.2 g.) in 35 cc. C6H6 added to 14.2 g. VII in 80 cc. pentane with stirring and moderate cooling and the mixture stirred 1 h. gave 43.8 g. orange-red EDA complex, R2[Ph(PhCH2)N]Al ← NPh:-CHPh (XIV), decomposed 85°, ν 1600 cm.-1 Crystalline VIII treated with an equimolar amount VI also gave XIV. XIV decomposed in C6H6 with EtOH, H2O, and aqueous Na2CO3 followed by measure merit of the extinction in the region 27,000-30,000 cm.-1 showed that 50% of the VI added was present unchanged, and, therefore, bound as a complex. XIV in C6H6 boiled 5 h. resulted in a change of the red color to pale yellow. VII (28.4 g.) in 50 cc. C6H6 treated with 108.6 g. VI in 150 cc. C6H6 with stirring and cooling, the solution refluxed 5 h. (oil bath at 90-5°) (11.4 g. isobutene evolved), and the residual isobutene displaced by a current of argon resulted in formation of RAl(NPhCH2Ph)2; hydrolysis and cleavage with HCl of the VI (0.2 mol) still present gave 15.2 g. PhNH2. VII (14.2 g.) treated with 36.2 g. VI in 100 cc. xylene under cooling, the solution of XIV treated with 29 g. VI, and boiled 4 h. at 160° (oil bath) gave 9.1 g. isobutene. VIII (19.4 g.) treated with 11 g. IX in C6H6 gave 24.4 g. colorless EDA complex, R2[Ph(PhCH2)N]Al ← NHPhCH2Ph, decomposed 110-15° (sinters above 95°). I (19.5 g.) in 40 cc. pentane

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added dropwise to 7.1 g. VII gave 18.1 g. orange-red EDA complex, R2[(pMeC6H4)(PhCH2)N]Al ← N(: CHPh)C6H4Me-p, decompd. 126°. II (2 mol) treated with 1 mol VII in C6H6 at below 40° gave orange-yellow R2(PhCH2)2N]Al ← N(:CHPh)CH2Ph. VII (4.3 g.) in 10 cc. C6H6 treated with 15.4 g. Ph2C: NPh (XV) in 30 cc. C6H6, the mixt. stirred 2 h. at 40°, and the black-red soln. cooled to 5° and partially concd. (concn. increased formation of ppt.) gave 5.5 g. XV, m. 113°, which indicated that the complex had decompd. during isolation; the mother liquor dild. with C6H6, decompd. with EtOH and a little H2O, filtered from Al(OH)3, and evapd. in vacuo gave a gum, which yielded 7.3 g. Ph2CHNHPh, m. 85° (EtOH), after treatment with a little EtOH. VII (14.2 g.) in 15 cc. C6H6 treated gradually with 46.2 g. IV in 60 cc. hot C6H6 gave 8 g. orange-red R2[2-C10H7(PhCH2)N]Al ← N(:CHPh)C10H7-2, m. 40° (slight decompn.), decompg. in soln. X (21.2 g.) made into a paste with 10 cc. C6H6, treated with 19.7 g. V in 20 cc. hot C6H6, heated 3 h. at 60°, concd. in vacuo, and dild. with 30 cc. pentane gave 18.5 g. black-brown R2[αC10HT(α-C10H7CH2)N]Al ← N(: CHC10H7-α)C10H7-α, decompd. 98-100°, which gave a deep red color and partially decompd. in soln. Phenanthridine (35.8 g.) in 60 cc. hot C6H6 added dropwise to 14.2 g. VII gave 42 g. light red EDA complex, XI complexed with phenanthridine, m. 118° (slight decompn.). VII (14.2 g.) treated with 35.8 g. acridine in 85 cc. hot C6H6, and the mixt. kept 3 h. at 70° gave 38.8 g. dark brown EDA complex, diisobutyl-9,10-dihydroacridylaluminum complexed with acridine, decompd. 192°, giving a deep green C6H6 soln. with ν 15,900 cm.-1; concn. of the mother liquor gave 9.9 g. addnl. impure complex. XI (1 mol) treated with 1 mol XII in C6H6 and the soln. concd. gave the corresponding colorless EDA complex, decompd. 134-5° (reddens above 115°).

ACCESSION NUMBER:	1964:23471 CAPLUS
DOCUMENT NUMBER:	60:23471
ORIGINAL REFERENCE NO.:	60:4167c-h,4168a-d
TITLE:	Organometallic molecular compounds. I. Complexes of ethers and amines with organoaluminum amides
AUTHOR(S):	Neumann, Wilhelm P.
CORPORATE SOURCE:	Max-Planck-Inst. Kohlenforsch., Muelheim-Ruhr, Germany
SOURCE:	Ann. (1963), 667, 1-11
DOCUMENT TYPE:	Journal
LANGUAGE:	German
OTHER SOURCE(S):	CASREACT 60:23471

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AB Spectral studies were made on 0.01M solns. of the low-spin, purple complex (Ni(S2P(OEt)2)2 = Ni(dtp))2 mixed with various amines in the same solvent. With PhNH2, Ph2NH, and MeCN, the purple color is unchanged. Ethanolamines, NH2CH2CH2NH2, NH2CH2CH2CH2NH2, and gaseous NH3 give pale bluish green colors and violet decomposition products precipitate after a few hrs. Secondary amines (Bu2NH, iso-Bu2NH, Et2NH, piperidine, dicyclohexylamine, and dibenzylamine) give strong yellow or orange colors. This is attributed to the formation of a distorted 5-coordinate low-spin complex. Tertiary amines give about 20% of the yellow form. 2,2'-Bipyridine and o-phenanthroline give high-spin green crystalline compds. Absorption bands for the yellow adducts are tabulated.

ACCESSION NUMBER:	1963:401522 CAPLUS
DOCUMENT NUMBER:	59:1522
ORIGINAL REFERENCE NO.:	59:215h,216a
TITLE:	Adducts of nickel(II) diethyldithiophosphate with secondary amines and heterocyclic diimines
AUTHOR(S):	Joergensen, Chr. Klixbull
CORPORATE SOURCE:	Cyanamid European Res. Inst., Cologny, Switz.
SOURCE:	Acta Chemica Scandinavica (1963), 17, 533-5 CODEN: ACHSE7; ISSN: 0904-213X
DOCUMENT TYPE:	Journal
LANGUAGE:	English

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AB 5-(Disubstituted amino)-1,296,3,4-thiatriazoles (I) containing groups of varying electronegativities to prevent a possible tautomeric shift were synthesized via N,N-disubstituted thiocarbamoyl chlorides (II) and from 4,4-disubstituted thiosemicarbazides (III). The III were prepared from II and from thioglycolic acids. The II were prepared by the dropwise addition of
0.05-0.24 moles thiophosgene in 50 ml. Et2O over 45 min. to 0.1-0.48 moles appropriate secondary amine in Et2O at less than 5°. Filtration and concentration of the reaction mixture gave II recrystd. from CHCl3 and petr.
ether. In this manner N,N-diethyl-(IV), N-methyl-N-phenyl-(V), N-ethyl-N-phenyl-(VI), and N,N-dibenzylthiocarbamoyl chlorides (VII) were prepared in 38-60% yield. The N,N-dimethyl compound, however, was prepared by
Billiter's [Ber. 37, 4319 (1904)] method of direct thiophosgenation of dimethylamine hydrochloride in the presence of NaOH. Variation of the moles of NaOH and temperature gave yield of 1.6-50% N,N-dimethylthiocarbamoyl
chloride (VIII). Extraction of the mother liquor with CHCl3 gave tetramethylthiuram monosulfide which also was obtained by treating tetramethylthiuram disulfide with KCN. The preparation of III from II was accomplished by the addition of 0.02-0.11 mole of the appropriate II to 0.044-0.22 mole hydrazine at 0-5° in Et2O over 30 min. and recrystg. the precipitated solids from absolute EtOH. The compds. prepared were:
4,4-dimethyl-(IX), m. 156-7°; 4,4-diethyl-(X), m. 84-5° 4-methyl-4-phenyl-, m. 122.5°; 4-ethyl-4-phenyl-, m. 119°; and 4,4-di-benzylthiosemicarbazide, m. 139.5°. The p-nitrobenzaldehyde derivs. of the thiosemicarbazides were: 4,4-diethyl-, m. 174° 4-methyl-4-phenyl-, m. 141-3; 4-ethyl-4-phenyl-, m. 139.5° and 4,4-dibenzyl-, m. 161.2°. The reaction of 0.062 mole hydrazine hydrochloride in anhydrous tetrahydrofuran and 0.02 mole VI gave the thiosemicarbazide of VI and 3% 4,4'-diethyl-4,4'-diphenyl-1-carbiminyl thiosemicarbazide, m. 157°. The same products were obtained when Et2O was used as the solvent but when Me2CO was used as solvent the product was an unidentified viscous red oil. The preparation of I
from II was accomplished by treating 0.1 mole NaN3 in 50 ml. H2O with 0.05 mole of the appropriate II 30 min., allowing to cool to room temperature 12-24
hrs., extracting with Et2O, concentrating the Et2O, and recrystg. the products from
absolute EtOH. With VIII the reaction mixture was heated at 100° 2 hrs. and gave 5-(dimethylamino)-1,2,3,4-thiatriazole, m. 51°. Reaction temperature and time had considerable effect when NaN3 was treated with VI; at
60-70° for 0.5 hr. the product was 20% 5-(ethylphenylamino)-1,2,3,4-thiatriazole, m. 148.5-9.5°; at 28° for 12 hrs. only an unidentified oil was obtained; at 50° for 10 hrs. the product was an unidentified solid; and at 100° for 1 hr. the products were S and H2S. The reaction of NaN3 and VII at 50° for 6 hrs. gave 50% 5-(dibenzylamino)-1,2,3,4-thiatriazole, m. 89-90°. The reaction of NaN3 and V gave 45% 5-(methyl-phenylamino)-1,2,3,4-thiatriazole, m. 56.5°. The preparation of N,N-(disubstituted-thiocarbamoyl)thioglycolic acids was accomplished by treating, at less than 15°, a mixture of 1.1 moles appropriate secondary amine and 1.0 mole KOH in 100 ml. H2O and 150 ml. EtOH with 1.0 mole CS2 followed by 1.0 mole chloro-acetic acid neutralized with 1.0 mole KOH. Acidification and filtration gave: N,N-dimethyl-(XI), m. 144-6°, N,N-diethyl-(XII), m. 89°,

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N,N-dibutyl-, m. 69°, or N-methyl-N-phenyl-thiocarbamylthioglycolic acid, m. 199-200°. When a mixt. of 1 mole XI or XII with 1.2 moles NaOH and 1.2 moles hydrazine (as hydrate, 90%) was refluxed 6 hrs. it gave 66% IX or 48% X, resp. In the case of XII the yield of X was 12% at 3 hrs. and 20% at 20 hrs. The benzaldehyde derivs. of IX and X m. 162° and 174°, resp. An appropriate III was converted to its counterpart I by treating 0.09 mole III with 0.1 mole HCl at 5° with 6.9 g. NaNO2 in 15 ml. H2O, removing the ppt. after 75% of the NaNO2 was added, and adding the remaining NaNO2 soln. to the filtrate to a reddish-yellow color. This method gave 5-substituted 1,2,3,4-thiatriazoles (substituent given); 80% 5-amino, m. 128-30°; 63% 5-methylamino, m. 93-6°; 89% 5-anilino (XIII), m. 142-5°; and 30% 5-(dimethylamino), m. 51° (XIV). The prepn. of 5-chloro-1,2,3,4-thiatriazole (XV) was done by treating 0.031 mole NaN3 in 100 ml. H2O with 0.031 mole thiophosgene at -5° over 30 min. and filtering under N. The yield was 94%. A larger scale prepn. using 0.197 mole reactants was satisfactory; however, when 2 moles NaN3 per mole thiophosgene was used the reaction exploded violently even when packed in ice. The reaction of 0.01 mole XV with a slight molar excess of dimethyl-amine in H2O at -5° for 30 min. gave 50% XIV. In a similar manner aniline in EtOH added to XV gave 40% XIII. Equimolar amts. XV and dibenzylamine in Et2O gave 35% 5-(dibenzylamino)-1,2,3,4-thiatriazole, m. 90°. Pyrolytic decompn. studies of the thiatriazoles prepd. was done by heating at 90° a uniform mixt. of 0.0015 mole of the compd. with 3 g. Ottawa sand and measuring the vol. of N evolved over a period of time. This revealed a decreasing order of stability as (Me2N > H2N > (C6H5CH2)N (sic) > PhHN > MeHN showing that the 5-(monosubstituted) compds. are less stable than I. The synthesis of 5-hydrazino-1,2,3,4- thiatriazole and 5-azido-1,2,3,4-thiatriazole failed due to their instability. The prepn. of XIV from XI and NaN3 failed. The prepn. of N,N-disubstituted thiocarbamoylthioglycolic acid from the following secondary amines failed; diisopropyl-, ethylphenyl-, diphenyl-, and dicyclohexylamine. Instead of the expected diisopropyl-, dibutyl-, or diphenyl-substituted II the prepn. gave only S, H2S, or an unidentified oil. The prepn. of 4,4-diisopropyl-, 4,4-dibutyl-, 4-methyl-4-phenyl-, or 4-ethyl-4-phenylthiosemicarbazide also failed. The prepn. of 5-(diethylamino)-1,2,3,4-thiatriazole from II gave only an oil contg. S and N, or the oil and free S. From the appropriate thiosemicarbazides the following I could not be prepd. (substituents given): diethyl, diisopropyl, dibutyl, methylphenyl, or ethylphenyl. XV did not react successfully with Ph2NH, N2H4, PhNHNH2, or NaNO2. The reaction of VII with NaN3 in Me2CO, EtOH, or with hydrazine in Me2CO gave tetramethylthiuram monosulfide. There was no reaction of VII with XIV in EtOH or with NH3 in Et2O and with aniline in Et2O an unidentified product was obtained. The reaction of IV with hydrazine in tetrahydrofuron also gave unidentified products. The reaction of IV with NaN3 in dimethylform-amide gave an unidentified yellow solid, in. 123°. XIV did not react with MeI or with HCl in EtOH. The reaction of carboxy methyldithiocarbohydrazide K salt with NaN3gave a reaction mixt. which with benzaldehyde gave benzal azine, m. 95°, or with 1-naphthaldehyde gave 1-naphthal azine, m. 152°. Diazotization of thiocarbohydrazide gave an unstable unidentified product, m. 223°. The reaction of benzaldehyde carboxymethyldithio-carbohydrazone with NaN3 in NaOH soln. gave only the starting material.

ACCESSION NUMBER: 1963:27258 CAPLUS
DOCUMENT NUMBER: 58:27258
ORIGINAL REFERENCE NO.: 58:4543g-h,4544a-b,4545a
TITLE: Thiatriazoles-azido and thio groups attached to the same carbon atom

L17 ANSWER 10 OF 49 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)
AUTHOR(S): Lieber, Eugene; Lawyer, Cornelius B.
CORPORATE SOURCE: DePaul Univ., Chicago
SOURCE: United States Department of Commerce, Office of Technical Services, PB Report (1962), 154,269, 108 pp. CODEN: XCPRAL; ISSN: 0099-8567
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

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AB cf. C.A. 53, 15000h. Degradation via oxidative alkali melts gives insight into the hardening of PhOH with (CH2)6N4, e.g. bonding occurs mainly in the o-position of PhOH with formation of dibenzylamines and chains, while bonding in the p-position occurs only after prolonged heating and higher temps. 2,2'-Dihydroxy-3,3',5,5'-tetramethyldibenzylamine (I) and tris(2-hydroxy-3,5-dimethylbenzyl)amine (II) are easily converted to hydroxytrimesic acid (III) by use of an oxidative alkali melt with PbO2 which rapidly degrades the CH2-N bridges, but under the same conditions 2,2'-dihydroxy-3,3',6,6'-tetramethylbenzylamine (IV) and 2,2'-dihydroxy-4,4',6,6'-tetramethylbenzylamine (V) undergo decarboxylation, IV to 2-hydroxyisophthalic acid (VI), and V to 2-hydroxyterephthalic acid (VII) and 5-hydroxyisophthalic acid (VIII). The degradation of xylenol-(CH2)6N4 condensates IV and V via oxidative alkali melts proceeds along unknown paths and leads to products from whose constitution the structure of the starting materials cannot be determined with certainty, but the degradation of
PhOH-(CH2)6N4 condensates proceeds without side reaction, e.g. o-hydroxybenzylamine (IX) and 2,2'-dihydroxydibenzylamine (X) form salicylic acid (XI), 4-hydroxybenzylamine, 4,4'-dihydroxydibenzylamine, and the tribenzylamine (XII) yield p-hydroxybenzoic acid (XIII). The three-ring compds. 2,6-bis(2-hydroxybenzylaminomethyl)phenol (XIV) and 2,6-bis(4-hydroxybenzylaminomethyl)phenol (XV) are synthesized by dehalogenation of 2,6-bis(acetylaminomethyl)-4-chlorophenol (XVI) with Raney Ni to 2,6-bis(acetylaminomethyl)phenol (XVII), saponification of XVII to
2,6-bis(aminomethyl)phenol (XVIII), which with o-, and p-HOC6H4CHO, resp., forms the three-ring azomethine from which is formed XIV and XV by catalytic hydrogenation. Via oxidative alkali melts XIV is split into XI and VI, and XV into XI and VI. The separation of the acids is worked out preparatively, also the paper chromatography of the phenol carboxylic acids. The PhOH-(CH2)6N4 rosins are prepared by hardening PhOH and (CH2)6N4 in 3:2 mole ratio at various temps, and reaction times. PhOH and (CH2)6N4, on hardening at 100°, combine almost exclusively in the o-position with the formation of X and o-substituted chains of the type XIV. Only on oxidative degradation of rosins which are hardened longer at 100° and above can the formation of XVII be observed, which supposes the formation of p-compds. But here too, the o-compds. XI and VI constitute the main yield. Hardening at 180° of a condensate which forms at 100° by a three-dimensional bonding with NH3 splitting off forms III through oxidative degradation. Through oxidative degradation are affected not only CH2-N bridges, but also CH2 bridges. The PhOH-(CH2)6N4 condensates obtained at 100-30° contain mainly CH2-N bridges, as shown by N values, while those obtained at 180° contain CH2 bridges besides, although the position of the bridges cannot be determined
by the results. PhOH-(CH2)6N4 condensate (2 g.) is mixed intimately with 9-11 g. PbO2 and introduced portionwise with good stirring into a melt of 40 g. KOH and 10 g. H2O at 320°, cooled, carefully diluted with 50 ml. H2O, acidified with 50% H2SO4, made alkaline, the precipitated PbSO4 separated and
washed well, the filtrate acidified again, extracted several times with ether,
the ether dried, evaporated, and the residue treated with superheated steam to
yield XI. The residue is extracted with hot H2O, VI crystallizing out of the
filtrate. The residue contains XII. III is obtained by evaporating the aqueous

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phase after Et2O sepn. and extn. of the evapd. residue. Oxidn. of I yields 76% III and of II, 75% III. Yields of VI from IV and VII and VIII from V are small. On paper chromatography the following results are obtained with S & S 2043a/gl, descending in 80:4:16 EtOH-concd. aq. NH3-H2O, 1% FeCl3 soln. as developer (acid, RF, color of spots, and ultraviolet fluorescence given): XI, 0.75, blue, strongly blue; XIII, 0.57, weakly yellow, -; VII, 0.50, blue, strongly light blue; 4-hydroxyphthalic acid, 0.41, violet, weakly blue; VI, 0.31, pink, dark blue; VIII, 0.25, -, strongly yellow; III, 0.12, yellow-brown, blue. p-ClC6H4OH (60 g.) is dissolved in 150 ml. satd. alc. HCl and treated with methylolacetamide (from 70 g. AcNH2 and 35 g. paraformaldehyde), HCl gas added 24 hrs. under ice cooling, the pptg. XVI.HCl sepd., taken up in H2O, and XVI liberated by dil. NH3 in 60% yield, m. 202° (40% EtOH). XVI (6 g.) in 100 ml. EtOH, 3 ml. H2O and 0.9 g. NaOH is hydrogenated in the presence of 10 g. Raney Ni till H absorption ceases, neutralized, the solvent evapd. in vacuo, and the residue recrystd. from H2O several times to yield XVII, prisms, m. 175°, yield 80%. Over 30 g. XVII is poured 50 ml. EtOH and 150 ml. HCl (d. 1.19), and with addn. of HCl 6-8 hrs. refluxed, cooled, and satd. with HCl gas to ppt. XVIII.HCl, long spears, m. 215° (decompn.). XVIII.HCl (11.5 g.) is dissolved in 100 ml. EtOH, and boiled 30 min. with 12.5 g. o-HOC6H4CHO and 8.6 g. NaHCO3. On cooling, the azomethine (XIX), yellow needles, m. 187° (xylene), seps. XIX (2 g.) is dissolved in 50 ml. EtOH and 3 ml. HCl (d. 1.19) and hydrogenated with a PtO2 slurry (100 mg. PtO2 in 20 ml. EtOH). Evapn. yields hygroscopic crystals of XIV.HCl, from which is obtained XIV (decompn. from 180°) through NaHCO3 treatment. In the same manner XV is obtained by treatment of XVIII with p-HOC6H4CHO and NaHCO3 to form the azomethine, weakly yellow needles, m. 183°, which is then reduced to XV.HCl, hygroscopic needles, and XV, decomp. from 160°, liberated by NaHCO3 treatment.

ACCESSION NUMBER: 1960:22796 CAPLUS
DOCUMENT NUMBER: 54:22796
ORIGINAL REFERENCE NO.: 54:4442f-i,4443a-h
TITLE: The structure of artificial resins. VII. Oxidative degradation of the methylene-nitrogen bridges in phenol-hexamethylenetetramine condensates
AUTHOR(S): Zigeuner, G.; Jellinek, K.
CORPORATE SOURCE: Univ. Graz, Austria
SOURCE: Monatshefte fuer Chemie (1959), 90, 232-8
CODEN: MOCMB7; ISSN: 0026-9247
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L17 ANSWER 12 OF 49 CAPLUS COPYRIGHT 2005 ACS on STN
AB cf. C.A. 52, 6041e. A dilute Et2O solution of TiBr4 added to an amine solution gave ppts. containing 1 mole of the bromide to 4 of the following amines (color and m.p. of the derivs. in parentheses): propylaniline (108°, brown-gray), butylaniline (97°, white-gray), isoamylaniline (144°, dirty white), dibenzylamine (-, white), di-(p-tolyl)amine (182-3°, gray-white), dipropylamine (300°, white), N,N'-dimethyl-p-phenylenediamine (-, dark ash), N,N-dimethyl-o-toluidine (86°, pink-gray), N,N-diethyl-o-toluidine (90°, gray-white), N,N-dimethyl-p-toluidine (78°, yellow), N,N-diethyl-p-toluidine (156°, dirty white), triethylamine (309-10°, dirty white), γ-picoline (212°, white), tribenzylamine (214°, white), p,p'-bismethylaminobenzo)phenone (-, orange-yellow).

ACCESSION NUMBER: 1959:55131 CAPLUS
DOCUMENT NUMBER: 53:55131
ORIGINAL REFERENCE NO.: 53:9877f-g
TITLE: Amino derivatives of titanium tetrabromide. IV
AUTHOR(S): Prasad, Sarju; Tripathi, Jai Beniprasad
CORPORATE SOURCE: Banaras Hindu Univ., Varanashi
SOURCE: J. Indian Chem. Soc. (1958), 35, 415-18
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L17 ANSWER 13 OF 49 CAPLUS COPYRIGHT 2005 ACS on STN
AB The following compds. were prepared by addition of an ethereal solution of the amine to NiI2 in ether. The products were analyzed to determine composition (amine = A, formula, color, m.p.) 1-naphthylamine, NiA4I2, green, 101°; 2-naphthylamine, NiA4I2, green, 20°; p-toluidine. NiA4I2, blue-grey, 22°; benzylamine, NiA4I2, blue-pink, liquid; benzidine, NiA2I2, blue, 102°; o-dianisidine, NiA2I2, blue-green, 164°; o-phenylenediamine, NiA2I2, blue, 168° decompose; p-phenylenediamine, NiA2I2, blue, 260°; o-tolidine, NiA2I2, blue-grey, 240°; phenylhydrazine, NiA2I2, yellow, 18°; diphenylamine, NiA4I2, green, 158°; dibenzylamine, NiA4I2, blue-green, liquid; Et3N, NiAI2, yellow, 174°; Et3N, NiA4I2, yellow, 179°; diethylaniline, NiA4I2, blue, liquid; piperazine, NiA4I2, blue-green, 210° decompose; piperidine, NiA4I2, yellow-green, 139°.

ACCESSION NUMBER: 1959:15956 CAPLUS
DOCUMENT NUMBER: 53:15956
ORIGINAL REFERENCE NO.: 53:2920h-i,2921a
TITLE: Compounds of nickel iodide with amines and heterocyclic basis
AUTHOR(S): Prasad, Sarju; Krishnan, V.
CORPORATE SOURCE: Banaras Hindu Univ., Varanasi
SOURCE: J. Indian Chem. Soc. (1958), 35, 352-4
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L17 ANSWER 14 OF 49 CAPLUS COPYRIGHT 2005 ACS on STN
AB (Ph(CH2)1-3]2NXN-R1R2 (X = alkylene which can be substituted; R1and (or) R2 = H, alkyl, or alkylene forming a ring) are prepared by conventional methods. They combine high musculotropic action with a strong neurotropic spasmolytic effect. Thus, 22.2 g. β-piperidinoethyl chloride, 33.8 g. bis(β-phenylethyl)amine, and 20 g. K2CO3 was refluxed in EtOH 20 hrs., allowed to cool, filtered, distilled in vacuo, the fraction, b8 190-230°, dissolved in dilute HCl, filtered, and treated with aqueous Na2CO3 until the mono-HCl salt of N-(β-piperidinoethyl)-bis(βphenylethyl)amine, m. 169-70° (EtOH-Et2O), separated Also prepared were: N-(β-diethylaminoethyl)bis(β-phenylethyl)amine [HCl salt, m. 173-5° (EtOH); di-MeI salt, m. 210-11° (decomposition) (EtOH); MeI salt, m. 92-3° (EtOAc)]; N-(γ-piperidinopropyl)-N-dibenzylamine, b4 154-6° (oxalate, m. 158°).

ACCESSION NUMBER: 1959:7153 CAPLUS
DOCUMENT NUMBER: 53:7153
ORIGINAL REFERENCE NO.: 53:1385e-g
TITLE: Tertiary basically substituted aralkyamines with musculotropic and neurotropic spasmolytic action
INVENTOR(S): Pfanz, Hermann; Breslauer, Henri; Jassmann, Edgar
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DD 12188		19561009	DD	

L17 ANSWER 15 OF 49 CAPLUS COPYRIGHT 2005 ACS on STN
AB Dilute Et2O solns. of amines were added to FeI2 in Et2O with shaking until precipitation was complete, the precipitate filtered and washed with anhydrous Et2O until
the washing did not produce a precipitate with FeI2. In this manner were prepared
the following FeX2I2[X, color, and m.p. (decomposition) given]:
p-MeC6H4NH2, dark brown, 150°; α-C10H7NH2, light brown.
165°; β-C10H7NH2, black, 147°; Me2C6H3NH2,
yellow-brown, 215°; PhNH2, dark brown, 140°; p-EtOC6H4NH2,
grey, 220°; m-MeC6H4NH2, muddy, 197°; p-EtZNC6H4NH2,
black, 210°; o-MeC6H4NH2, black, 183°; PhCH2NH2,
brown, 230°; o-MeOC6H4NH2, brown, 228°;
o-EtOC6H4NH2, orange-brown, 189°; p-MeOC6H4NH2, brown,
216°; Me2CHNH2, reddish brown, 180°; the following FeXI2:
o-C10H6(NH2)2, black, 140°; [MeO(NH2)C6H3]2, green,
276°; (H2NCH2)2, dark brown, 118°; p-C6H4(NH2)2, black,
210°; [o-Me(NH2)C6H3]2, ash, 206°;
o-C6H4(NH2)2, black, 225°; PhNHNH2, white, 155°;
(p-H2NC6H4)2, yellow-brown, 219°; and the following FeX3I2: Ph2NH,
brown, 224°; PhNHCH2Ph, yellow-brown, 256°; PhNHEt, black,
213°; (p-MeC6H4)2NH, yellow-brown, 222°; (PhCH2)2NH, brown,
214°; Pr2NH, yellow-brown, 263°; PhNHPr, yellow-brown,
239°; (PhCH2)3N, brownish black, 264°; Et3N, brown,
215°; o-Me2NC6H4Me, yellow-brown, 211°. The compds.
are stable in a dry atmospheric at room temperature, but hydrolyze in contact with
moisture, Na2CO3, or NaOH solns. The compds. from monoamines hydrolyze
slowly at room temperature and rapidly at higher temps., giving Fe(OH)2; those
from diamines are more stable and hydrolyze slowly, even on boiling,
indicating that chelation has taken place.
ACCESSION NUMBER: 1958:34899 CAPLUS
DOCUMENT NUMBER: 52:34899
ORIGINAL REFERENCE NO.: 52:6226b-f
TITLE: Amino derivatives of ferrous iodide
AUTHOR(S): Prasad, Sarju; Krishnamurty, D. R.
CORPORATE SOURCE: Banaras Hindu Univ.
SOURCE: J. Indian Chem. Soc. (1957), 34, 563-7
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L17 ANSWER 16 OF 49 CAPLUS COPYRIGHT 2005 ACS on STN
AB cf. C.A. 51, 11150d. The amino derivs. (I) of TiBr4 with aromatic secondary and tertiary amines, [Ti(Am)4]Br4, were prepared by reactions between Et2O solns. of TiBr4 and of the respective amines. After 1 hr. of stirring, the ppts. were removed, washed with Et2O, and dried. Analyses (chemical and potentiometric) showed composition only, as % Ti, Br, and N. I were
prepared from these amines (color and m.p. of the derivs. in parentheses): N-methylaniline (light yellow, 236°); N-ethylaniline (gray white, 242°); N-benzylaniline (green, 167°); diphenylamine (yellowish white, 226°); N,N-di-methylaniline (light gray, 138° decompose); N,N-diethylaniline (white, 248°); quinoline (brownish gray, 122°); N-benzylideneaniline (yellow, 160°); N,N-dibenzylaniline (gray, 154°, formula [Ti(PhN(CH2Ph)2)4]Br4); p-amino-N,N-diethylaniline (black, 305°, formula [Ti(Et2NC6H4NH2)2]Br4). H2O, aqueous NaOH, and aqueous Na2CO3 initiate
hydrolysis of I to precipitate Ti(OH)4, but this is complete only at 50°. Heating with soda-lime frees the amine. I are generally insol. in organic solvents, but those containing Ph2NH, quinoline, N-benzylideneaniline, N,N-dibenzylaniline, and p-amino-N,N-diethylaniline dissolve in CHCl3, EtOH, and acetone.
ACCESSION NUMBER: 1958:14821 CAPLUS
DOCUMENT NUMBER: 52:14821
ORIGINAL REFERENCE NO.: 52:2636e-h
TITLE: Amino derivatives of titanium tetrabromide. II
AUTHOR(S): Prasad, Sarju; Tripathi, Jai Beniprasad
CORPORATE SOURCE: Banaras Hindu Univ.
SOURCE: J. Indian Chem. Soc. (1957), 34, 452-6
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L17 ANSWER 17 OF 49 CAPLUS COPYRIGHT 2005 ACS on STN
AB 2,5-Dimethoxy-1,4-benzoquinone (5 g.) and 20 cc. NH4OH refluxed 1 hr. in 200 cc. EtOH and cooled gave 3.3 g. 2,5-diamino-1,4-benzoquinone (I), glistening violet needles, m. 328-30° (decomposition). I (0.5 g.) refluxed 0.5 hr. with 1 g. NaOAc in 5 cc. Ac2O and cooled gave I diacetate, yellow needles, m. 272° (decomposition). I (0.5 g.), 2 g. K2CO3, and a few drops of BzCl refluxed 8 hrs. in 70 cc. dry Me2CO, filtered, and evaporated, and the residue crystallized from glacial AcOH gave 0.25
g. I dibenzoate, pale orange needles, m. 258°. I (0.5 g.) heated about 0.5 hr. with 10 cc. Ac2O, 2 g. Zn dust, and 1 g. NaOAc, diluted with 10 cc. glacial AcOH, heated 10 min., and cooled gave 0.5 g. I tetraacetate, long needles, m. 263° (decomposition). MeOCS2K (from 1.8 g. KOH in 30 cc. MeOH and 5 cc. H2O and 2 g. CS2) heated 15 hrs. on the H2O bath with 0.5 g. I, treated with C, cooled, and filtered, the filtrate heated to boiling and diluted with about 5 cc. AcOH, and the crystalline precipitate
repptd. from 5% alc. KOH with AcOH yielded 0.3 g. dimercaptobenzodiazole, yellow needles, m. above 400°. I and 4 equivs. of the appropriate aldehyde refluxed about 5 hrs. in absolute EtOH containing a few drops of pyridine
and cooled, and the precipitate recrystd. from glacial AcOH gave the corresponding 2,6-disubstituted benzodioxazoles (substituents, color of product, m.p., and % yield given): Ph, cream-yellow, 325°, 70; p-MeC6H4, colorless, 325-8°, 75; p-MeOC6H4, light pink, 315-17°, 82; o-ClC6H4, pale yellow, 263°, 72; o-HOC6H4, colorless, 340°, 38. The 3,6-di-Cl derivative of I gave similarly the following 2,6-disubstituted-4,8-dichlorobenzodioxazoles (same data given): Ph, cream-yellow, 332°, 50; p-MeC6H4, cream-yellow, 320°, 62; p-MeOC6H4, cream-yellow, 310-12°, 40; o-ClC6H4, light yellow, 308-10°, 65. Very pure 2,5-dihydroxy-1,4-benzoquinone (II) (0.5 g.) treated with a few drops of alc. NH3 precipitated 0.25 g. di-NH4 salt of II, decomposed at 170° without melting; an aqueous solution acidified gave II, m. 212-14°. II (0.2 g.) in 30 cc. dry C6H6 heated 10 hrs. with a few drops PhCH2NH2 in a sealed tube at 100° gave 0.18 g. dibenzylamine salt of II, changed to brown at 140° and then decomposed without melting. 2,5-Dimethoxybenzoquinone (III) (0.3 g.) in 30 cc. absolute EtOH refluxed with
the appropriate alkylamine (a few drops) during 20 min. gave the corresponding 2,5-bis(alkylamino)-1,4-benzoquinone (alkyl group, color of crystals, and m.p. given): Et, brilliant crimson, 210°; Bu, bronze, 160°; PhCH2 (IV), deep red glistening (orange in H2SO4), 252°. PhNH2 gave similarly during 5 hrs. the Ph analog, did not melt up to 350°. III (0.2 g.) in 50 cc. dry C6H6 containing a few drops PhCH2N2 under N in a sealed tube exposed 3 days to sunlight deposited 0.22 g. IV, m. 252°. IV (0.3 g) refluxed 7 hrs. with a few drops BzH in absolute EtOH in the presence of piperidine and cooled
gave only unchanged IV.
ACCESSION NUMBER: 1957:51834 CAPLUS
DOCUMENT NUMBER: 51:51834
ORIGINAL REFERENCE NO.: 51:9597c-h
TITLE: Benzodioxazoles
AUTHOR(S): Osman, Abdel-Meguid
CORPORATE SOURCE: A'in Shams Univ., Abassia, Cairo, Egypt
SOURCE: Journal of the American Chemical Society (1957), 79, 960-8
CODEN: JACSAT; ISSN: 0002-7863
DOCUMENT TYPE: Journal

L17 ANSWER 17 OF 49 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)
LANGUAGE: Unavailable
OTHER SOURCE(S): CASREACT 51:51834

L17 ANSWER 18 OF 49 CAPLUS COPYRIGHT 2005 ACS on STN
AB Color is developed by use of bromocresol purple (I) with phosphate buffer (pH 5.2). The method is sensitive to as little as 2.5 γ dibenamine (I)/ml. urine or 5.0 γ dibenamine alc./ml. urine. In the concentration range 2.5-40.0 γ I/ml. there is conformance to the Lambert-Beer law. To the 10 ml. solution to be tested is added 5 ml. Sorenson phosphate buffer (pH 5.2), 5 ml. 0.8% alkaline I solution, and 50 ml. benzene. The mixture is shaken 2 min. and the aqueous phase removed and shaken with 50 ml. benzene. The combined benzene exts. are filtered and shaken twice with 10 ml. 0.05N NaOH. The colored NaOH exts. are filtered and the volume made up to 25 ml. with 0.05N NaOH.

ACCESSION NUMBER: 1957:2291 CAPLUS
DOCUMENT NUMBER: 51:2291
ORIGINAL REFERENCE NO.: 51:532f-h
TITLE: The estimation of dibenamine and dibenamine-like compounds in biological mixtures
AUTHOR(S): Hofmann, H.; Boltze, K. H.; Weyland, D.
CORPORATE SOURCE: Friedrich Schiller Univ., Jena, Germany
SOURCE: Experientia (1956), 1, 362-3
DOCUMENT TYPE: Journal
LANGUAGE: German

L17 ANSWER 19 OF 49 CAPLUS COPYRIGHT 2005 ACS on STN
AB Heat 20 g. of sample in a dry, 200-ml. silica digestion flask until the oil begins to fume, allowing the vapors to be swept away by a strong draught. Heat until only 1 or 2 ml. remains. Cool, add 3-3.5 ml. of pure concentrated H_2SO_4 and then 2-3 ml. of concentrated HNO_3. Heat with addition of $HClO_4$ or a little more HNO_3 if necessary. Cool, add 10 ml. of water, and again heat to fuming. Dilute to 50 ml. in a separatory funnel, add 1 ml. of 5% Na_2SO_3 solution to remove traces of nitrous fumes and treat with 10 ml. of CCl_4 and one of the following color reagents: Zn dibenzyldithiocarbamate, dibenzylamine salt of dibenzyldithiocarbamate, dibenzyldithiocarbamic acid, K dibenzyldithiocarbamate. Filter the lower layer through a plug of cotton wool and measure the optical d. at 435 mμ. Good results were obtained in determining 0.4-12.0 γ of Cu. All 4 coloring agents are equally efficient.

ACCESSION NUMBER: 1955:3180 CAPLUS
DOCUMENT NUMBER: 49:3180
ORIGINAL REFERENCE NO.: 49:645b-d
TITLE: Determination of copper in oils and fats by means of dibenzyldithiocarbamic acid and its salts
AUTHOR(S): Abbott, D. C.; Polhill, R. D. A.
CORPORATE SOURCE: Clement's Inn Passage, London
SOURCE: Analyst (1954), 79, 547-50
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L17 ANSWER 20 OF 49 CAPLUS COPYRIGHT 2005 ACS on STN
AB The colors obtained with 20 aromatic dialkylated bases and o-toluenesulfochloride, with and without the addition of glacial AcOH, are listed and can serve to help identify the bases. Five procedures are given: (1) Treat the sample with 10 ml. AcOH, shake, and allow to stand 3 min. Then add quickly 6 drops of perhydrol and from the resulting color estimate the probable type of base present. (2) After adding the AcOH heat for 5 min. in a paraffin bath at 140°. Remove the test tube from the bath, dip in toluene and then in MeOH and allow to cool to room temperature (3) From the solution of the base, evaporate off the ether, add 15 drops of toluene sulfochloride and after 30 sec. add 10 ml. of Ac_2O, shake, and heat 5 min. at 140°. (4) After heating 8 min. with Ac_2O at 140°, add 15 drops of toluene sulfochloride and heat 4 min. more at 140°. (5) Instead of perhydrol in the above test, add 0.2 g. PbO_2, stopper with a cork and shake vigorously 30 times, wait one min., then shake another 30 times. Filter and eventually dilute with Ac_2O.

ACCESSION NUMBER: 1951:41039 CAPLUS
DOCUMENT NUMBER: 45:41039
ORIGINAL REFERENCE NO.: 45:6970g-i,6971a
TITLE: Detection and determination of dialkylated aromatic bases
AUTHOR(S): Wurzschmitt, Bernhard
CORPORATE SOURCE: Badische Anilin- u. Soda-Fabrik, Ludwigshafen a. Rhein, Germany
SOURCE: Zeitschrift fuer Analytische Chemie (1951), 133, 17-27
CODEN: ZANCA8; ISSN: 0372-7920
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L17 ANSWER 21 OF 49 CAPLUS COPYRIGHT 2005 ACS on STN
AB The perfectly colorless shells of chestnuts (Castanea vesca) harvested before they are ripe assume a dull brown color after some hours in the air, owing to the presence of d-catechol (I), which was isolated in about 0.6% yield by immediately heating the shells 1 hr. at 75° in alc. to destroy the enzymes, decanting the alc. (II) (later found to contain the greater part of the I), drying the shells (150 g.) in the air and in vacuo, grinding, extracting several times with 500 cc. absolute alc., concentrating the exts. in vacuo to a thin sirup, removing the rest of the solvent in a desiccator, extracting several times with water at 50°, concentrating the exts. to 70 cc. in vacuo, extracting with benzene and then exhaustively with ether, repeating the extraction with ether after the water layer had been concentrated to half its volume, evaporating the ether exts., drying in a desiccator, dissolving in 15 cc. dry acetone, slowly treating, with vigorous stirring, with 90 cc. benzene (which mostly precipitated the impurities, but also some I, as a sirup), evaporating the Me_2CO-C_6H_6 solution in vacuo, dissolving in 10 cc. hot water, and clearing with talc; in some hrs. 200 mg. I separated in pink needles; the Me_2CO-C_6H_6 purification repeated twice more on the 1st Me_2CO-C_6H_6 precipitate yielded another 100 mg. I. The 1st alc. solution (II), similarly treated, gave 600 mg. I. Recrystn. of the combined crude I from water gave 800 mg. I.$4H_2O$, m. 93-5°, losing 19.93% in weight over P_2O_5 at 55° and 17 mm. and then m. 174.5-5°, $[\alpha]_{20}D$ 14.4 ± 1° (in 1:1 Me_2CO-H_2O); pentaacetate, m. 131-2°, $[\alpha]_{20}D$ 38.5° ($CHCl_3$).

ACCESSION NUMBER: 1949:6352 CAPLUS
DOCUMENT NUMBER: 43:6352
ORIGINAL REFERENCE NO.: 43:1341b-f
TITLE: Natural tannins. I. Tannins of the chestnut. 1. The occurrence of catechol in chestnut shells
AUTHOR(S): Schmidt, Otto Th.; Hull, Georg
SOURCE: Chemische Berichte (1947), 80, 509-10
CODEN: CHBEAM; ISSN: 0009-2940
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L17 ANSWER 22 OF 49 CAPLUS COPYRIGHT 2005 ACS on STN
AB There are added to the products small quantities of slightly volatile monoamines, the color of which is fast to light, and which contain at least one C6H6 ring but no O or S, e. g., rayon fabric which has been delustered with TiO2 is treated with an aqueous solution containing 1-10% of its weight of N,N-dimethyl-o-toluidine; or alternatively, the TiO2 is preliminarily treated with a 3% aqueous suspension of dibenzylamine.

ACCESSION NUMBER: 1945:5266 CAPLUS
DOCUMENT NUMBER: 39:5266
ORIGINAL REFERENCE NO.: 39:822a-b
TITLE: Improving the properties of manufactured products and coatings containing TiO2 and reprecipitated cellulose
PATENT ASSIGNEE(S): I. G. Farbenindustrie AG
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
BE 446011		19420731	BE	

L17 ANSWER 23 OF 49 CAPLUS COPYRIGHT 2005 ACS on STN
AB Color formation with H2SeO3-H2SO4 solns. is not a specific reaction of phenolic compds. Many N compds., especially those containing 2 or 3 aromatic nuclei, give intense color reactions with this reagent. Place 1 mg. of the compound on a spot plate and add a drop of a 0.5% solution of H2SeO3 in concentrated H2SO4. Carry out a similar test simultaneously with H2SO4 alone and observe the color changes. Of a total of 108 compds. studied the following gave decided color changes in the reagent solution but not in the H2SO4 alone (sensitivities in γ are given in parentheses for some compds.): o,p-aminobiphenyl, 4-aminodiphenylamine-HCl (0.5), aniline, benzeneazodiphenylamine (0.1), p-bromoaniline, carbanilide, m-chloroaniline, cholesterol, cysteine-HCl, 2,4-diaminodiphenylamine (1.0), dibenzylaniline, s-dimethylcarbanilide, di-2-naphthylamine (0.1), di-p-phenetylurea, diphenylamine (10.0), diphenylbenzidine (1.0), diphenylcarbamine Cl (7.0), s-diphenylcarbazide (1.0), s-diphenylcarbazone, s-diphenylethylenediamine, 4,5-diphenylglyoxalone (0.5), diphenylpiperazine (10.0), 1,4-diphenylsemicarbazide (0.1), 4,4-diphenylsemicarbazide (50.0), diphenylthiocarbazide (0.1), s-di-(o,p)-tolylthiourea, s-di-(o, m, p)-tolylurea, formyl diphenylamine (10.0), methylthiocarbanilide (30.0), leucine, methyldiphenylamine (10.0), (1,2)-naphthylamine, 4-nitrodiphenylamine (0.1), p-nitrophenylhydrazine, phenylthiourea, thiocarbanilide (1.0), tolidine (2.0), (o,p)-toluidine-HCl, triphenylguanidine, tryptophan. The colors produced by 1- and 2-naphthylamine and di-2-naphthylamine can be readily distinguished. The test for opium alkaloids with this reagent is not conclusive unless interfering phenols and N compds. are known to be absent.

ACCESSION NUMBER: 1942:24523 CAPLUS
DOCUMENT NUMBER: 36:24523
ORIGINAL REFERENCE NO.: 36:3750b-f
TITLE: Color reactions of organic nitrogen compounds with selenious acid-sulfuric acid solutions
AUTHOR(S): Dewey, Bartlett T.; Gelman, Albert H.
SOURCE: Industrial and Engineering Chemistry, Analytical Edition (1942), 14, 361-2
CODEN: IENAAD; ISSN: 0096-4484
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L17 ANSWER 24 OF 49 CAPLUS COPYRIGHT 2005 ACS on STN
AB cf. C. A. 33, 1284.9. o-IC6H4CHO and MeNO2 in Et3N give 65-70% of α-nitro-β-(2-iodophenyl)ethylene (I), pale yellow, m. 113-14°; fuming HNO3 gives α-nitro-β-(6-iodo-3-nitrophenyl)ethylene (II), pale yellow, m. 145-6°. I and Br give an oil on treatment with warm EtOH-AcOK; fuming HNO3 gives a yellow compound, C8H4BrIN2O4, m. 136-7°; it gives an addition compound with p-MeC6H4NH2 but was not investigated further. The previous procedure was used for preparing the addition compds. of II, which were crystallized from EtOH; they are yellow or orange-yellow and are deeper in color than I; β-derivs. of α-nitro-β-(6-iodo-3-nitrophenyl)ethane: anilino, m. 115-16°; o-, m- and p-toluidino, m. 168-70°, 113-14° and 130-2°; o-, m- and p-anisidino, m. 146-8°, 140-2° and 123-4°; phenylhydrazino, m. 142-4°; β-naphthylhydrazino, m. 143-4°; hydroxylamino, m. 103-5°; semicarbazido, m. 187-8° (the last 2 are colorless) II in. C6H6, saturated with NH3 and allowed to evaporate spontaneously, gives α,α'-di(6-iodo-3-nitrophenyl)-β,β'-dinitrodiethylamine, m. 113-14°. II is the most active nitrostyrene thus far studied.

ACCESSION NUMBER: 1940:18285 CAPLUS
DOCUMENT NUMBER: 34:18285
ORIGINAL REFERENCE NO.: 34:2805e-g
TITLE: Action of aromatic amines on 3-nitro-6-iodonitrostyrene
AUTHOR(S): Worrall, David E.; Benington, Frederick
SOURCE: Journal of the American Chemical Society (1940), 62, 493-4
CODEN: JACSAT; ISSN: 0002-7863
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L17 ANSWER 25 OF 49 CAPLUS COPYRIGHT 2005 ACS on STN
AB cf. C. A. 32, 2115.4. Details are given of compds. of sym-C6H3(NO2)3 (I) and picric acid (II) with carbostyril and its derivs. and various quinolones and quinolines. The most striking variation in the tendency for complex formation with I is provided among the C-methylcarbostyrils by the unique failure in this respect of the 6-Me derivative; this appears to be constitutional and is contrary to the usually helpful influence of such substituents in amines or hydrocarbons; N-methylation of carbostyrils appears to reduce the probability of isolating homogeneous crystalline derivs. of I. The picrates obtained are manifestly "salt-like" in character if compared with the I complexes in color and m. p.; moreover they are frequently of different (i. e., 1:1) composition Their similar ease of preparation and moderate solubility in alc. suggests that the picrates of carbostyrils are not differentiated from 2-quinolone picrates as salts of "2-hydroxyquinolines," unless perhaps in the case of carbostyril picrates itself. These picrates may therefore be "H bond" adducts -NRC:O . . . HOX, stabilized by resonance. Picrates assumed to be "salt-like" in structure are indicated by the use of II as a suffix. Carbostyril (III) in EtOH gives the complex I.2III, S-yellow needles, m. 178°, and III.II, yellow needles, m. 132° (prepared in Et2O or from very concentrated solns. in MeOH or EtOH). Thiocarbostyril (IV) in EtOH gives the complex I.IV, light-brown plates, m. 163-5° and IV.II, crimson needles, m. 145°. Dihydrocarbostyril (V) yields the complex I.2V, yellow plates, m. 137-8°. The 3-Me derivative (VI) of III yields the complex I.2VI, light-yellow needles, and II.2VI, golden-yellow prisms, both with incongruent m. ps. The 4-Me derivative (VII) of III yields the complex I.2VII, canary-yellow prisms, m. 226-7° and VII.II, light-yellow needles, m. 164-5°. 4-Methyl-2-thiocarbostyril (VIII) in CHCl3 gives the complex I.2VIII, brown-yellow prisms, m. 190-2°, and 2VIII.II, orange-red plates, m. 193-5°. The 5-Me derivative (IX) of III m. 222-3°; it forms a complex I.2IX, light-yellow needles, m. 222-3°, and IX.II, yellow prisms, m. 156-7°. The 6-Me derivative (X) of III forms the complex X.II, pale-yellow needles, m. 171-2°. The 6-Me isomer (XI) of VIII forms a complex I.2XI, orange prisms, m. 159-61° (in CHCl3), and scarlet prisms with II (composition not determined), m. 140-2°. The 7-Me derivative (XII) of III m. 192-3°; it forms a complex I.2XII, canary-yellow needles, m. 203-4°, and XII.II, light-yellow needles, m. 163°. The 8-Me derivative (XIII) of III forms the complex I.2XIII, golden-yellow needles, m. 181°, and XIII.II, light-yellow needles, m. 128-9°. The 4,6-di-Me derivative (XIV) of III yields the complex I.2XIV, golden-yellow prisms with an incongruent m. p., and XIV.II, canary-yellow needles, m. 188°. The 4,7-di-Me derivative (XV) of III forms a complex I.2XV, S-yellow needles, m. 213-14°, and XV.II, light-yellow needles, m. 189-91°. The 4,8-di-Me derivative (XVI) of III gives a complex I.2XVI, S-yellow needles, m. 199-200°, and XVI.II, canary-yellow needles, m. 192-4°. 1-Methyl-2-quinolone (XVII) gives a complex I.XVII, light-yellow laminated plates, m. 77-9°, and XVII.II, yellow needles, m. 128-9°. 1-Methyl-2-thioquinolone (XVIII) yields the complex I.2XVIII, orange needles, m. 98-9°, and II.2XVIII, orange prisms, m. 104°. 1,6-Dimethyl-2-quinolone (XIX) yields the complex XIX.II, canary-yellow needles, m. 150°. The 1,7-isomer (XX) of XIX (pale yellow, m. 107-8°) gives a complex I.XX, pale yellow needles, m. 106-7°, and XX.II, lemon-yellow prisms, m. 132°. The 1,8-isomer (XXI) of XIX gives the complex XXI.II, canary-yellow needles, m. 134°. 2-Methoxyquinoline (XXII) forms the complex I.XXII, yellow plates, m. 89-90°, and XXII.II, yellow needles, m.

L17 ANSWER 25 OF 49 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)
170-1°. 2-Methylthioquinoline (XXIII) gives the complex I.XXIII, deep-yellow needles, m. 99-100°, and XXIII.II, yellow plates, m. 183-4°. 2-Methoxy-6-methylquinoline (XXIV) yields the complex I.XXIV, greenish yellow prisms, m. 72-3°, and XXIV.II, greenish yellow plates, m. 181-2°. The compd. XXIII.II was first obtained from IV and Me picrate (XXV) in MeOH; that it is not a mol. compd. follows from the synthesis by bubbling MeSH through MeONa in MeOH, adding 2-chloroquinoline in MeOH, boiling 2 h. and adding II. XI and XXV in boiling MeOH give 2-methylthio-6-methylquinoline picrate, golden-yellow plates, m. 196-7°. XVIII and XXV, boiled 10 min. in MeOH, give 2-methylthio-1-methylquinolinium picrate, deep-yellow plates, m. 175°; 1,6-dimethyl-2-thioquinolone was recovered unchanged even after 2 h. boiling. Crystn. of I from 6-methylquinoline gave the binary compd., pale-yellow needles, m. 63-5°; the 8-isomer afforded an analogous product, pale yellow with incongruent m. p. 2-Chloro-7-methylquinoline, m. 81° (picrate, canary-yellow plates, m. 113-14°). 3-Methylquinoline oxide-HCl, m. 192-4° (picrate, greenish yellow needles, incongruent m. p.). 6-Methylquinoline oxide-HCl, m. 172-3° (picrate, pale-yellow needles, m. 174-5°). 1,6-Dimethyl-2-thioquinolone, yellow, m. 137°. I and dibenzyl-o-toluidine give relatively lightly colored EtOH solns. which pptd. only the constituents; melts of these compds. in the proportions 1:1, 1:2 or 2:3 give viscous red liqs., disintegrated to colorless powders. Dibenzyl-o-toluidine picrate, canary-yellow prisms, m. 120-1°. Dibenzyl-m-toluidine (XXVI) and I in concd. EtOH soln. give a compd. 2I.3XXVI, ruby-red prisms, m. 71-2°; a soln. contg. the reactants in the ratio of 2:3 gives successive crops of complex until reduced to dryness: the picrate of XXVI, yellow prisms, m. 126-7°. The p-isomer (XXVII) of XXVI and I (2:1 in EtOH) give the complex I.2XXVII, ruby-red needles, m. 62-4°; the picrate of XXVII, golden-yellow plates, m. 174-5°. I and 1-thiocoumarin in concd. C6H6 or EtOH soln. give colorless solns. which pptd. only the components; the picrate, yellow needles, m. 148°; trans-o-aminocinnamic acid gives a binary complex with I, brick-red needles, m. 131°. I and 2-thiocoumarin in EtOH give the binary complex, light-brown plates, m. 87°. 2,4,5-Trinitrotoluene and (4-Me2NC6H4)2CH2 give a binary complex, dark-red needles, m. 92-3°; 2,4,6-(O2N)3C6H2Me and p-C6H4(NO2)2 did not afford cryst. derivs. The m.-p. curves are given for I with VII, X and XVII.

ACCESSION NUMBER: 1940:10524 CAPLUS
DOCUMENT NUMBER: 34:10524
ORIGINAL REFERENCE NO.: 34:1665f-i,1666a-i,1667a-b
TITLE: Complexes of polynitro compounds. III. Compounds of polynitro substances with derivatives of carbostyril, etc.
AUTHOR(S): Kent, Andrew; McNeil, Donald; Cowper, Robert M.
SOURCE: Journal of the Chemical Society, Abstracts (1939) 1858-62
CODEN: JCSAAZ; ISSN: 0590-9791
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

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AB For quant. determination dissolve 0.1 g. of veritol (I) in 10 cc. H2O and 15 cc. EtOH and titrate with 0.1 N NaOH, using phenolphthalein; add rosolic acid and titrate with 0.1 N H2SO4 to yellow. Both titrns. must be identical if the substance is pure. The factor per cc. is 0.0428. Differentiation from hordenine (II), tyramine (III) and tyrosine (IV) was tried, making use of 22 different reagents; but most gave identical reactions. The following color reactions may be used: Cl water and NH3 give with I red, with II light yellow, with III yellow with green fluorescence, with IV red. HIO3 gives with I and III red, with II and IV neg. H2O2 with I and III red, with II and IV neg. Tyrosinase gives with I, III and IV red, with II neg. Colorimetric estns. of veritol may be effected with the diazo reaction, using either sulfanilic acid or p-nitroaniline, or with Wavelet's reagent, which gives a blue color in the presence of NH3.

ACCESSION NUMBER: 1940:6210 CAPLUS
DOCUMENT NUMBER: 34:6210
ORIGINAL REFERENCE NO.: 34:997b-e
TITLE: Chemistry of p-hydroxyphenylisopropylmethylamine or veritol
AUTHOR(S): Bonino, Rosa C. D'Alessio de Carnevale
SOURCE: Semana Medica (1939), II, 1314-23
CODEN: SEMEAS; ISSN: 0370-9590
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L17 ANSWER 27 OF 49 CAPLUS COPYRIGHT 2005 ACS on STN
AB cf. C. A. 32, 8420.7. Kostanecki's 1st method for the synthesis of flavones involving treatment of o-acetoxychalcone dibromides with alc. alkali has, hitherto, not been applicable for the synthesis of the numerous natural flavones containing a phloroglucinol nucleus, since the corresponding chalcone dibromides give benzylidenecoumaranones only on treatment with alc. alkali. The observation that o-hydroxychalcone dibromides in general give flavones when they are heated above the m. p. or are treated with alc. KCN has made possible the synthesis of III, V and VI from the corresponding chalcone dibromides. Phloroacetophenone tri-Me ether (5 g.) in 40 cc. Ac2O, treated in the cold with 40 cc. HI(d. 1.7), gives 4.8 g. of the 4,6-di-Me ether; AlCl3 gives 30% less product. 5-Bromo-2-hydroxy-4,6-dimethoxyphenyl α,β-dibromo-β-phenylethyl ketone (I), yellow, m. 186°, results in 7 g. yield from 10 g. of 2-hydroxy-4,6-dimethoxyphenyl styryl ketone and Br in CS2 at 0°; I or its Ac derivative (II), heated at 195° and 7 mm., gives 6-bromo-5,7-dimethoxyflavone which with HI in Ac2O (refluxing 2 h.) yields chrysin (III). I or II with hot C5H5N gives 4-bromo-3,5-dimethoxy-1-benzylidenecoumaran-2-one, m. 251°, which also results with hot or cold 10% NaOH in EtOH or Me2CO (Kostanecki and Tambor, Ber. 32, 2260(1899) give 223°). The α,β-dibromo-β-p-anisylethyl homolog (IV) of I, yellow, m. 165°; heating above the m. p. at 7 mm. gives 6-bromo-5,7,4'-trimethoxyflavone, yellow, m. 250°; HI in Ac2O gives apigenin (V). IV with 10% aqueous NaOH gives 4-bromo-3,5-dimethoxy-1-anisylidenecoumaran-2-one, yellow, m. 243°; heated with C5H5N for 10 min., IV yields 5-bromo-2-hydroxy-4,6-dimethoxyphenyl p-methoxystyryl ketone, orange, m. 184-5°; it gives a dark color with EtOH-FeCl3 and a yellow color with H2SO4. The α,β-dibromo-β-3,4-dimethoxyphenylethyl homolog of I, orange, m. 165°; heating at 190° under reduced pressure gives 6-bromo-5,7,3',4'-tetramethoxyflavone, yellow, m. 258°; a better yield results by heating 2 h. with excess EtOH-KCN; HI gives luteolin (VI).

ACCESSION NUMBER: 1939:17115 CAPLUS
DOCUMENT NUMBER: 33:17115
ORIGINAL REFERENCE NO.: 33:2498h-i,2499a-d
TITLE: Chalcones: A new synthesis of chrysin, apigenin and luteolin
AUTHOR(S): Hutchins, W. A.; Wheeler, T. S.
SOURCE: Journal of the Chemical Society, Abstracts (1939) 91-4
CODEN: JCSAAZ; ISSN: 0590-9791
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L17 ANSWER 28 OF 49 CAPLUS COPYRIGHT 2005 ACS on STN
AB Halogen-containing derivs. of rubber, gutta-percha, balata and synthetic rubber such as methylbutadiene (polyhaloprenes such as polymerized chloroprene being excluded) are milled, with or without solvents with basic materials that retard their decomposition under heat and mech. treatment. These may be oxides of Ca, Sr, Ba, Mg, Al, Ni, Zn, Co, Ti, Sn, Sb or Pb, Ba(OH)2, carbonates of Ba, Ca, Sr, Mg, Na or guanidine, or **dibenzylamine**, NH2Am, (CH2)6N4, diphenylethylenediamine, benzylamine, NHPh2, benzylaminophenol, benzalaminophenol, tetramethyldiaminodiphenylmethane, diphenylguanidine phthalate, guantol or dibenzylaniline. To the composition there may be added during milling: (1) rubber age retarders, (2) plasticizers, (3) fillers, (4) pigments or dyes, (5) natural or synthetic rubber, (6) hardeners. Sheets calendered from the milled mixture may vary in color from transparency to black. The mixture may be molded under heat and pressure.

ACCESSION NUMBER: 1938:31862 CAPLUS
DOCUMENT NUMBER: 32:31862
ORIGINAL REFERENCE NO.: 32:4381f-h
TITLE: Halogenated rubber
PATENT ASSIGNEE(S): Marbon Corp.
DOCUMENT TYPE: Patent
LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
GB 476733		19371209	GB	

L17 ANSWER 29 OF 49 CAPLUS COPYRIGHT 2005 ACS on STN
GI For diagram(s), see printed CA Issue.
AB Of the possible substituted NH4 dithiocarbamates, the literature contains alkylammonium N-alkyldithiocarbamates and dialkylammonium N-dialkyldithiocarbamates with like alkyl groups, i.e., SC(NHR)SNH3R and SC(NR2)SNH2R2, and of the possible mixed substituted NH4 dithiocarbamates, alkylammonium N-alkyldithiocarbamates, dialkylammonium N-alkyldithiocarbamates and alkylammonium dialkyldithiocarbamates, i.e., SC(NHR)SNH3R', SC(NHR)SNH2R'2 and SC(NR2)SNH3R' are known. On the contrary, alkylammonium and dialkylammonium dithiocarbamates of the type: SC(NH2)SNH3R and SC(NH2)SNH2R2 and dialkylammonium dialkyldithiocarbamates with differing alkyl groups, i.e., SC(NR2)SNH2R'2, are still unknown. The present paper describes the preparation of these unknown dithiocarbamates, with particular attention to SC(NH2)SNH2R2 compds., one object of which was to study their behavior with aldehydes in connection with previous expts. in the same field (cf. C. A. 26, 1251). The results show that alkylammonium and dialkylammonium dithiocarbamates can be prepared from concentrated aqueous SC(NH2)SNH4 (I) and soluble salts of the primary and secondary amines. Similarly, SC(NR2)SNH2R'2 compds. were prepared from NH4 N-dialkyldithiocarbamates and secondary amine salts. SC(NH2)SNH3R and SC(NH2)SNH2R2 compds. are unstable, whereas SC(NR2)SNH2R'2 compds. are as stable as the already known SC(NHR)SNH3R and SC(NR2)SNH2R2 types. More complex dithiocarbamates of other organic bases were also prepared, as well as alkyl and dialkylammonium trithiocarbonates of the SC(SNH3R)2 and SC(SNH2R2)2 types, by the reaction of SC(SNH4)2 with soluble salts of primary and secondary amines. These trithiocarbonates are less stable than the dithiocarbamates. The new dithiocarbamates were treated with HCHO and AcH, and the results are of interest in connection with earlier expts. on the reaction of other dithiocarbamates with aldehydes (cf. Ann. 65, 43; 168, 232; Ann. chim. [7], 9, 119(1898); Levi, C. A. 24, 830, 3994). Dialkylammonium dithiocarbamates do not react with HCHO, whereas with AcH they form derivs. of the type: SC(N:CHMe)SN(:CHMe)R2. With HCHO and with AcH, alkylammonium dithiocarbamates form condensation products containing 2 aldehyde residues per mol. of dithiocarbamate, the constitution of which is uncertain, but which is either SC(N:CHR)SN(:CHR)HR or SC.NH.CHR.N(:CHR)R.S. With HCHO, SC(NHR)SNH2R'2 compds. form condensation products containing 1 aldehyde residue per mol. of dithiocarbamate, the formula of which is either SC(NHR)SNCH2R'2 or SC.NR.CH2.NHR'2.S. With AcH the condensation products are liquids, which were not investigated further. Exptl.-The precipitate from a mixture of cold concentrated aqueous I and PhCH2-NH3Cl (II), washed successively with water, EtOH and Et2O and recrystd. from EtOH, yields monobenzylammonium dithiocarbamate, SC(NH2)SNH3CH2Ph (III), stable, m. 90-3° (decomposition). Prepared in a similar way, camphylammonium dithiocarbamate, C11H22N2S2, has a pearly luster, and m. 100-4° (decomposition). With I, aqueous salts of primary aliphatic amines do not precipitate, even when concentrated, the corresponding dithiocarbamates, but the latter are probably formed and remain in solution Other new dithiocarbamates include the following: Diethylammonium, C5H14N2S2, m. 98-105° (decomposition). Dipropylammonium, C7H18N2S2, m. 80-90° (decomposition). Diisobutylammonium, C9H22N2S2, m. 83-93° (decomposition). Piperidonium, C6H14N2S2, m. 80-90° (decomposition). Dibenzylammonium, C15H18N2S2, m. 145-55° (decomposition) (because of the low solubility of II in water, it must be prepared in hot water), more stable than the preceding compds.

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Methylphenylammonium, C8H12N2S2, m. imperfectly below 100° (it cannot be crystd. from EtOH because in hot EtOH it decomps. with pptn. of I), unstable and liberates H2S, s-Diphenylguanidine (IV), prepd. from cold concd. aq. solns. of I and HN:C(NHPh)2. HCl, with crystn. from boiling water, straw-color, m. 98-100° (decompn.). as-Diphenylguanidine, prepd. like IV (though cryst., no m. p. is given). It was found that the method of Paulson (cf. U. S. Pat. 1,575,865) for prepg. HN:C(NH2)NPh2 is better than that of Arndt and Rosenau (C. A. 12, 1187). With EtOAc as solvent, a good yield of HN:C(NH2)NHPh is also obtained. s-Di-o-tolylguanidine, prepd. like IV, pale straw color, m. 130-2° (decompn.). s-Triphenylguanidine, m. 88-90°. It has a tendency to sep. as a pitch, both in the original reaction and in the final recrystn. from water, but on standing the pitches become cryst. as-Triphenylguanidine, m. 103-6° (decompn.). Quinine, prepd. by adding excess concd. aq. I to hot, almost satd. quinine-HCl, and recrystg. the pitch (after solidification) from boiling water, m. 107-9° (to a yellow liquid). Quinidine, after solidification of the pitch, and recrystn. from boiling water, m. 202-5° (to a brown-red liquid). Cinchonine, ppts. directly in cryst. form, m. 208-9° (to a brown-red liquid). Strychnine, does not m. up to 250°. Brucine, m. approx. 140°. Dimethylammonium pentamethylenedithiocarbamate, crystd. from EtOH, m. 84-6°. Diisobutylammonium dimethyldithiocarbamate, m. 84-6°. With NH2Et2Cl, NH2Pr2Cl and C5H10NH2Cl, concd. aq. SC(NMe2)SNH4 does not ppt. the corresponding dithiocarbamates, and under the same conditions with NH2Et2Cl, NH2Pr2Cl and primary aliphatic amines, SC(NHC5H10)SNH4 does not ppt. the corresponding dithiocarbamates. Quinine pentamethylenedithiocarbamate (V). Quinine dimethyldithiocarbamate (VI). Strychnine pentamethylenedithiocarbamate (VII). Strychnine dimethyldithiocarbamate (VIII). Though V, VI, VII and VIII are cryst. no m. ps. are given. The ppt. from a mixt. of concd. aq. SC(SNH4)2 and concd. aq. II, washed successively with water, EtOH and Et2O, yields monobenzylammonium trithiocarbonate, SC(SNH3CH2Ph)2 (IX), stable for a few hrs. after its prepn. (though it loses traces of H2S), but after several hrs. it decomps. at an increasing rate; in cold water it gives the rose color which is characteristic of trithiocarbonates. Prepd. like IX, dipropylammonium trithiocarbonate, SC(SNH2Pr2)2, is a rose color, it is so unstable that it had to be analyzed wet; its cold aq. solns. are a rose color. NH2Et2Cl and NH2Me2Cl do not ppt. the corresponding trithiocarbonates even in concd. solns. With (iso-Bu)2NH2Cl and its homologs, the free bases (not the trithiocarbonates) ppt. AcH (1.5 g.) added to cold aq. SC(NH2)SNH2Et2 (2.5 g. in 10 cc.), let stand, filtered (after about 15 min. the yellow suspension resinifies, so it is best to filter several times as soon as more ppt. forms), washed with Et2O and recrystd. from Et2O, yields diethylethylideneammonium ethylidenedithiocarbamate SC(N:CHMe)SN(:CHMe)Et2 (X), m. 82-3°, decomps. rapidly when let stand, but can be kept in a desiccator for a long time. Prepd. like X, dipropylethylideneammonium ethylidenedithiocarbamate, C11H22N2S2 (XI), recrystd. from Et2O, m. 81-2°, does not show such a strong tendency to resinify during its prepn. as does X, decomps. slowly when let stand several weeks. Prepd. like X and XI (there is no tendency to resinify), diisobutylethylideneammonium ethylidenedithiocarbamate, C13H26N2S2, m. 101°, is more stable than XI. III and HCHO form a pitch which, allowed to crystallize and then recrystd. from Me2CO, yields the condensation product, C10H12N2S2, m. 130°. It is either SC(N:CH2)SN(CH2Ph)H:CH2 or SC.NH.CH2.N(CH2Ph)(CH2).S. III and AcH form a flocculent ppt. which, crystd. from Me2CO, yields the condensation product, C12H16N2S2, m. 98°. SC(NHPr)SNH2Me2 and HCHO form a

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product which, dissolved in Et2O and repptd. by petr. ether, yields the condensation product, C7H16N2S2, m. 52°. It is either SC(NHPr)SN(:CH2)Me2 or SC. NPr. CH2. NMe2H. S.

ACCESSION NUMBER: 1932:18198 CAPLUS
DOCUMENT NUMBER: 26:18198
ORIGINAL REFERENCE NO.: 26:1902d-i,1903a-i
TITLE: Alkyl and dialkylammonium dithiocarbamates and trithiocarbonates, and dialkyl-alkylideneammonium alkylidenedithiocarbamates
AUTHOR(S): Levi, T. G.
SOURCE: Gazzetta Chimica Italiana (1931), 61, 803-14
CODEN: GCITA9; ISSN: 0016-5603
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L17 ANSWER 30 OF 49 CAPLUS COPYRIGHT 2005 ACS on STN
AB Amine hydrosulfides, prepared from amines and H2S in the absence of O2 or air, undergo rapid oxidation upon exposure to air. Those derived from the more volatile amines leave an almost quant. deposit of S; those from the less volatile amines are oxidized to the corresponding thiosulfates. These oxidation reactions take place without evidence of polysulfide formation. A mechanism is suggested for the oxidation reaction which fully accounts for the facts observed. Using a special apparatus, the following amine hydrosulfides were prepared (2 m. ps. are given in an open and a closed tube): Me, m. 40-4°, 90-2°; di-Me, m. 34-40°, -; tri-Me, m. 15-20°, 28-30°; Et, m. 50-5°, 55-7°; di-Et, m. -, 55-62°; tri-Et, m. 25-7°, -; Pr, m. 38-42°, 40-2°; di-Pr, m. 58-62, 76-8°; Bu, m. 18-20°, -; di-Bu, m. 25-30°, 28-32°; iso-Am, m. 62-7°, -; dibenzyl, m. 32-4°, -. The solubility in H2O decreases and the stability increases with increasing mol. weight The freshly prepared aqueous solns. precipitate CdS and PbS from the acetates; the aqueous solns. become yellow on standing and will dissolve free S, taking on a blood-red color indicative of polysulfide formation. Oxidation of iso-AmNH3SH in the air gives isoamylamine thiosulfate, m. 192-6°; Bu derivative, m. 180-93° (decomposition).

ACCESSION NUMBER: 1931:37671 CAPLUS
DOCUMENT NUMBER: 25:37671
ORIGINAL REFERENCE NO.: 25:4219g-i
TITLE: Sulfur derivatives of the simple amines. I. Amine hydrosulfides
AUTHOR(S): Achterhof, Marvin; Conaway, Rollin F.; Boord, Cecil E.
SOURCE: Journal of the American Chemical Society (1931), 53, 2682-8
CODEN: JACSAT; ISSN: 0002-7863
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

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AB Eleven new tests are proposed for the detection of the OCN ion. They are: (1) Add AlCl3 solution to a hot solution of KNCO; Al(OH)3 is precipitated (2) Add FeCl3; a reddish color is obtained, or Fe(OH)3 is precipitated when hot, accompanied by evolutions of gas. (3) CrCl3, gives a Cr(OH)3 precipitate These 3 reactions require a 2% solution of cyanate, while the reagents should contain 0.5% metal. (4) Add a few cc. of Ni(NO3)2 or NiSO4, then a few drops of pyridine to the KNCO solution; avoid an excess of reagent; blue [NiPy](NCO)2 ppts. immediately, or after a few hrs. when the solution is very dilute; 0.01 g. KNCO can be detected. (5) Co++ salts give blue [Co(NCO)4]K2 with as little as 0.02 g. cyanate. For smaller concns. add one drop of Co(NO3)2 in Me2CO to one drop of tested solution on a watch glass; a blue coloration is observed at the time the two drops meet, providing 0.0004 g. cyanate is present. (6) To the solution, add Co(NO3)2, then pyridine; pink crystals of [CoPy4)(NCO)2 precipitate with as little as 0.001 g. of cyanate. (7) To a 2% cyanate solution, add a few cc. Zu(NO3)2 solution, then pyridine until the precipitate no longer redissolves. Avoid an excess of cyanate, which redissolves [ZnPy2](NCO)2. (8) Add 1 cc. CuSO4 and 1-3 drops picoline: if a large quantity of cyanate is present, blue [Cu(C6H7N)2](NCO)2 ppts.; otherwise add 2-3 cc. CHCl3 and shake, obtaining a blue coloration in CHCl3. (9) Add 2-3 cc. **dibenzylamine** in AmOH (3 cc. amine per 10 cc. AmOH), then 2-3 cc. of 1% CuSO4, and rotate the test tube slowly; the alc. layer is colored violet by cyanate; 0.0001 g. can be detected. (10) Add the cyanate solution to Cd(NO3)2 solution, precipitating colorless [Cd(NCO)3]K; this reaction detects 0.01 g. cyanate. (11) Add 2-3 cc. of 1% Cd(NO3)2 solution, then a few drops of pyridine, precipitating crystalline [CdPy2](NCO)3; 0.01 g. cyanate is detectable. The following reaction is proposed to detect Co: add 1-2 cc. of 4% KNCO solution freshly prepared, then one drop of concentrated AcOH. A blue color is obtained with as little as 0.00004 g. Co. If Me2CO is added (2-4 cc.) without stirring, the supernatant solution will color it blue with as little as 0.00002 g. Co. The following reaction is proposed to detect Co++ in the presence of Fe+++: add 2-4 cc. NH4Cl or NH4NO3 solution; boil; add 2-4 cc. of 4% KNCO solution; a gaseous evolution occurs and Fe(OH)3 ppts.; filter while hot; the filtrate is blue if Co is present; if colorless, add a little KNCO solution to compensate decomposition of the cyanate by boiling. As little as 0.0005 g. of Co will give a blue color. If Fe++ is present, it should be oxidized with HNO3, then neutralized with K2CO3 before testing with cyanate. Two new ammines have been prepared: [Cu(C4H2N)2](NCO)3, blue crystals from 2 g. CuSO4 in 100 cc. H2O and 2-cc. picoline in 50 cc. H2O; purified from alc. or CHCl3. [Cu(C14H15N)2](NCO)2, violet crystals from 4 g. CuSO4 in 100 cc. H2O + concentrated KNCO (enough for complete solution) and an emulsion of 3 cc. **dibenzylamine** in 100 cc. H2O, with efficient shaking; purification by recrystn. from Me2CO and washing with Et2O on the filter.
ACCESSION NUMBER: 1929:24669 CAPLUS
DOCUMENT NUMBER: 23:24669
ORIGINAL REFERENCE NO.: 23:2905c-i
TITLE: Metallic cyanates. VI. (1) New reactions of cyanic acid. (2) Qualitative test for cobalt. (3) New test for cobalt in the presence of iron

L17 ANSWER 31 OF 49 CAPLUS COPYRIGHT 2005 ACS on STN (Continued)
AUTHOR(S): Ripan, R.
CORPORATE SOURCE: Univ. Cluj
SOURCE: Buletinul Societatii de Stiinte din Cluj (1928), 4, 144-53
CODEN: BTUJAZ; ISSN: 0366-3868
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L17 ANSWER 32 OF 49 CAPLUS COPYRIGHT 2005 ACS on STN
AB If PhNH2, e. g., in a strongly acid solution containing NaSCN is treated in the cold with Br, the reaction 2NaSCN + Br2 = 2NaBr + (SCN)2, being ionic, proceeds so rapidly that the reaction PhNH2 + Br2 = BrC5H4NH2.HBr is negligible if an excess of NaSCN is used. The hydrolysis 3(SCN)2 + 4H2O = 5HSCN + H2SO4 + HCN is greatly retarded in the presence of the acid, and the same is true of the polymerization, so that under these conditions the reaction PhNH2 + (SCN)2 = p-NCSC6H4NH2 (I) + HSCN takes place. Numerous other substances have been successfully thiocyanated in this way. I, m. 57-8°, was obtained in 87% yield from 4.6 g. PhNH2 in 12 cc. of 96% AcOH and 25 g. NaSCN in 130 cc. AcOH treated with 5.09 cc. Br in 35 cc. AcOH; the AcOH mother liquors yielded 15% of 2,4(?)-dithiocyanoaniline, m. 198°. 1-C10H7NH2 gives no mono-NCS derivative; 2,4(?)-dithiocyano-1-naphthylamine, m. 204°, is obtained in 7 g. yield from 4.2 g. C10H7NH2, 17 g. NaSCN and 3.0 cc. Br in AcOH, while with 4.06 g. preformed (SCN)2 (from Pb(SCN)2 and Br) in Et2O, 10 g. C10H7NH2 yields 71% 4-thiocyano-1-naphthylamine, m. 146-7°, converted by standing in the air in alc. containing a few drops of NaOH into [1,4-C10H6(NH2)S-]2, m. 168°. 2-C10H7NH2 (7.15 g.) with 16 g. NaSCN and 2.5 cc. Br yields almost quant. 1-thiocyano-2-naphthylamine (II), sluters 150-4°, turns yellow, resolidifies and finally m. 261° (decomposition), converted into the amorphous [2,1-C10H6(NH2)S-]2 in alc. NaOH; with preformed (SCN)2 (0.5 mol.) in Et2O. 50% II is obtained. (p-NCSC5H4)2NH, m. 120°, is obtained in good yield from 0.2 g. Ph2NH, 2 g. NaSCN and the calculated amount of Br in 15% H2SO4. 1-C10H7OH (4.2 g.) with 9 g. NaSCN and 1.5 cc. Br in AcOH gives 70% 1,4-C10H6(OH)SCN, m. 113°, while 1 g. of the naphthol with 8 g. KSCN and 0.6 cc. Br yields 60% of 2,4(?)-dithiocyano-1-naphthol, faintly yellow, m. 118-9° (decomposition). 5,2-NCS(HO)C6H3CO2H, m. 167°, is obtained in 0.2 g. yield from 1.38 g. o-HOC6H4CO2H, 4 g. NaSCN and 1 cc. Br in HCO2H; the yield can undoubtedly be increased by changing the conditions. (CH2SCN)2, m. 90°, is obtained by passing C2H4 and Cl separately (care being taken that the C2H4 is always in excess) into NaSCN in cold AcOH, or by using Br in 15% HCl instead of Cl. Styrene (1 g.) with 2 g. NaSCN and 0.5 cc. Br in AcOH yields 65% of PhCH(SCN)CH2SCN, m. 101°; 2.8 g. anethole with 9 g. NaSCN and 1 cc. Br gives 75% of p-MeOC6H4CH(SCN)CH(SCN)Me, m. 87°, and from 1.2 g. antipyrine (III) and 2 g. NaSCN treated with Br in AcOH, the reaction mixture then being diluted with an equal volume of H2O and made faintly alkaline with 15% NaOH, is obtained 1.3 g. bis-[1-phenyl-2,3-dimethyl-5-pyrazolonyl] 4-disulfide, m. 256°, also obtained in 70% yield from 1.8 g. III and 5 g. NaSCN in AcOH treated with Cl until the mixture gave no red color with FeCl3 and then worked up as above.
ACCESSION NUMBER: 1926:12987 CAPLUS
DOCUMENT NUMBER: 20:12987
ORIGINAL REFERENCE NO.: 20:1603f-i,1604a-b
TITLE: New method for the thiocyanation of organic compounds
AUTHOR(S): Kaufmann, H. P.; Oehring, W.
SOURCE: Ber. (1926), 59B, 187-94
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
OTHER SOURCE(S): CASREACT 20:12987

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AB The fixation of Br upon PhCH: NPh was studied by Hantzsch in 1890 (Ber. 23, 2714). On adding a solution of Br to one of the base there is precipitated a pale yellow powder, PhCHBrNBrPh, m. 142° (decomposition). On contact with water it undergoes immediate decomposition to BzH and p-BrC6H4NH2. HBr. In contact with anhydrous solvents the color of the powder persists and a metal, as Cu or Au, if introduced, is converted into a bromide. With solvents containing water, the powder is decolorized-decomposition takes place as above and the metal is not attacked. Br addition products upon other Schiff bases, differing in the nature of the radicals of the aldehyde and of the base, are often very sensitive to moisture and do not always give very consistent results for the determination of Br. Isobutylideneaniline in anhydrous Et2O added to Br in C6H6 or CS2 gives a yellow powder evolving in moist air an irritating odor of Me2CBrCHO, not altered by reducing agents and does not set free Br with HBr. On contact with water, the principal reaction is decomposition into Me2CBrCHO + PhNH2. HBr. Benzylideneisobutylamine. The Br addition product, obtained as before, gradually forms a red-orange lower layer, slowly and incompletely forming ruby-red crystals, separating from CHCl3, anhydrous Et2O as a yellow crystalline powder, m. 83-4° (decomposition), has an irritating odor in moist air. With water, it decomps. into BzH + HBr + NHBrC4H9. Isobutylideneisobutylamine. Under the usual conditions there is obtained a thick red-orange liquid, which is very unstable. With water it decomps. into Me2CBrCHO + C4H9NH2. HBr. Benzylidenebenzylamine. The usual procedure gives in this case red crystals, m. 141-2°, slowly soluble in cold water with an irritating odor, becoming viscous on heating and giving off Br: PhCHBrNBrCH2Ph + H2O → HBr + BzH + NHBrCH2Ph; NHBrCH2Ph + HBr → Br2 + NH2CH2Ph. In conclusion, the decomposition of these Br derivs. by water is different according to the nature of the base and aldehyde that have produced the Schiff base. (1) One atom of Br passes into the amine nucleus when this is phenolic. The other yields HBr and the aldehyde is set free. (2) A brominated aldehyde is formed and a HBr salt of the base. (3) Br, being able to pass neither into the aldehyde group nor into the amine group, remains with the N in the form of a bromoamine. The other atom of Br yields HBr and the aldehyde is set free.
ACCESSION NUMBER: 1925:20343 CAPLUS
DOCUMENT NUMBER: 19:20343
ORIGINAL REFERENCE NO.: 19:2645c-h
TITLE: The bromine addition products of the Schiff bases
AUTHOR(S): Berg, M. A.
SOURCE: Bull. soc. chim. (1925), 37, 637-41
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

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AB cf. C. A. 18, 830. p-Ethylbenzyl alc., b9 115-7°, was prepared in 40% yield by shaking P-EtC6H4CHO (obtained in 15 g. yield from 100 g. PhEt, 100 g. C6H6, 125 g. AlCl3, 25 g. CuCl, CO and HCl) with concentrated KOH.
Several hrs.' heating with concentrated HCl gives the chloride, b11 81-2°. p-Phenylbenzyl alc., b11 183-4°, m. 101-2°; concentrated H2SO4 gives a bluish green color. Catalytic reduction of PhC6H4CN in 30% decalin solution by Ni and H gave a 70% yield of a mixture of
p-phenylbenzylamine, m. 127-8° (HCl salt, m. 282°; picrate, m. 205°; Ac derivative, m. 180°; Bz derivative, m. 162°; phenylthiourea, m. 150°; methiodide, m. 221°) and di-p-phenylbenzylamine, m. 132° (HCl salt, m. above 300°; NO compound, m. 1707°). NaNO2 gives nearly a quant. yield of the alc., from which, with concentrated HCl in a sealed tube, the chloride, m. 68°, is obtained. PhCH2NHMe is conveniently prepared by the reduction of the amine by Ni and H. With 2 mols. p-MeC6H4CH2Cl it gives a 70% yield of benzyl-p-methylbenzylmethylamine (I),b11 160° (methiodide, m. 190°). The corresponding p-Ph derivative (II), b9 190-2°, m. 44° (HCl salt, m. 187°; picrate, m. 146°; methiodide, m. 162°). p-Methylbenzylmethylamine, b11 83° (HCl salt, m. 174°; picrate, m. 145°), is obtained by reducing with Na and EtOH the condensation product, MeC6H4CH:NMe, b11 83°, obtained from p-MeC6H4CHO and MeNH2. p-Ethylbenzyl derivative (III), b9 181°. p-Phenylbenzyl derivative (IV), b13 253-5°. Butenyl derivative (V), b11, 116-8°. Cinnamyl derivative (VI), b12 218-20°. p-Ethylbenzyl-methylamine, b10 105°. p-Phenylbenzyl derivative (VII), b11, 255-7°. (HCl salt, m. 205°). PhC6H4CHO and MeNH2 give the Schiff base, PhC6H4CH:NMe, m. 51°, which is reduced by Na and EtOH to phenylbenzylmethylamine, b11 173-4° (70% yield); cinnamyl derivative (VIII), b10 220° (HCl salt, m. 224°). Cinnamylmethylamine, b12 110-2°, in 60% yield from MeNH2 and the chloride in C6H6. Allyl derivative (IX), b11 166-8°; crotonyl derivative (X), b10 180-2°. The action of BrCN on these bases gave a mixture of 3 products: the quaternary compound from the
base and the bromide which is split off (A);the bromide freed from the base by shaking with dilute HCl, was then combined with Me3N (B); and the cyanamide (C). X gave a compound A, C23H28NBr, m. 79°; B was formed in only small amts., as was C, crotonylmethylcyanamide, b55 92-3°. IX gave an oily A, which was transformed into the Cl derivative and then yielded a PtCl4 salt, C44H52N2Cl6Pt, m. 85°, B was pure cinnamyltrimethylammonium bromide, m. 165° and C methylallylcyanamide, b. 150°. I gave an addition compound of p-MeC6H4CH2Br and I, C24H28NBr, m. 184°, p-methylbenzyltriammonium bromide, m. 170-5°, and benzylmethylcyanamide (XI), b12 139-42°. III gave the compound C27H34NBr, m. 168°, containing 2EtC6H4CH2-groups, and p-ethylbenzyltrimethylammonium bromide, analyzed as the PtCl4 salt, m. 216°. In the case of VII, the product A was oily; phenylbenzyltrimethylammonium bromide (XII), m. 200°. II gave an oily A, XII and XI. IV gives an oily A, XII and a C containing Br. The pure methylbenzylmethylcyanamide b10 140-2°. VI gives a small amount of an oily A; methylbenzyltrimethylammonium bromide, m. 194°; and cinnamylmethylcyanimide, oily. V also gave an oily A, the same B as from I and crotonylmethylcyanamide, b45 80-5°. VIII gave an oily A, a B, C16H20NBr, m. 198°, and cinnamylmetlhylcyanamide, oily. The rate of reaction of EtONa upon various chlorides at 31.6° is expressed by the following values of k (time 12 hrs.): PhCH2Cl, 7.86; MeC6H4CH2Cl, 11.71; EtC6H4CH2Cl, 14.48; PhC6H4CH2Cl, 74.06. The relation

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of these results to the question of the firmness of attachment of the residues is discussed.
ACCESSION NUMBER: 1924:13572 CAPLUS
DOCUMENT NUMBER: 18:13572
ORIGINAL REFERENCE NO.: 18:1830f-i,1831a-c
TITLE: Firmness of attachment of organic residues. II
AUTHOR(S): v. Braun, Julius; Engel, Hans
SOURCE: Ann. (1924), 436, 299-320
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

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GI For diagram(s), see printed CA Issue.
AB (o-O2NC6H4CH2)2CAcCO2Et (6 g.) shaken in 21.5 g. SnCl2 in a warm mixture of 20 cc. AcOH and 20 cc. fuming HCl and heated 0.5 hr. on the H2O bath yields 4-5 g. of a Sn salt giving, when shaken in Et2O with KOH, the base C6H4 , sinters 178°, m. 184°; boiled with HI it splits off 1 mol. CO2, yielding a base, m. 165-7°, which is apparently impure II (see below). (o-O2NC6H4CH2)2C(CO2Et)2 is converted into the free acid, m. 149°, in 85% yield by heating 20 g. of it with 160 cc. H2SO4 (d. 1.83) and 80 cc. H2O 10-2 min. at 180-5°; this with 1 equivalent PCl5 gives di-[o-nitrobenzyl]acetyl chloride (I), m. 91-2°, 17.5 g. of this, allowed to stand 24 hrs. in 20 cc. C6H6 with a magma prepared from 2.3 g. Na powder allowed to stand 5 hrs. with 25 cc. each of C6H6 and CH2(CO2Et)2, gives di-Et [di-o-nitrobenzylacetyl]malonate, sinters 77°, m. 80°, gives a dark red color with FeCl3, and boiled 3 hrs. with 6 parts HCl changes, without dissolving, into di-[o-nitrobenzyl]acetone, m. 89-9.5°; 3 g. of this, refluxed 1 hr. with 15 cc. HI and 2 g. red P, yields 3-o-aminobenzylquinaldine (II), m. 166-7°, which forms diacid salts, evolves 1 mol. N2 with hot NaNO2-HCl, gives with C6H4(CO)2O at 300° a compound C25H18O2N2, yellow, m. 127-8°, and with BzH at 130° a yellow base, m. 170-1°. Allowed to stand overnight with 10 parts C6H6 and 1 part AlCl3, I gives di-[o-nitrobenzyl]acetophenone, m. 108-8.5°, reduced by HI-P to 2-phenyl-3-o-aminobenzylquinoline, m. 177-8°, which, fused with C6H4(CO)2O, yields a compound C30H20O2N2, m. 185°. With NH3 in C6H6, I gives di-[o-nitrobenzyl]acetamide, m. 162°, which could not be degraded by the Hofmann method to the amine; this, however, was obtained as follows: 5 g. of the amide in 5 cc. MeOH treated with 0.64 g. Na in 18 cc. MeOH and then with 2.5 g. Br in 5 cc. MeOH and boiled 10 min. gave Me di-[o-nitrobenzyl]methylcarbamate, m. 139°, 5 g. of which, heated 0.5 hr. at 120° with 16 cc. H2SO4 (d. 1.83) and 8 cc. H2O until there was no further evolution of CO2, yielded di-[o-nitrobenzyl]methylamine, m. 82-3°; this, boiled 45 min. with HI-P, yielded di-[o-aminobenzyl]methylamine.3HI, gradually decomps. above 230°; 3HCl salt, sinters above 260°; the free base is an oil solidifying to a glassy mass and soluble in H2O with alkaline reaction.
ACCESSION NUMBER: 1924:10950 CAPLUS
DOCUMENT NUMBER: 18:10950
ORIGINAL REFERENCE NO.: 18:1483g-i,1484a-c
TITLE: Some cyclic and aliphatic-aromatic bases from di-[o-nitrobenzyl]acetoacetic and -malonic esters
AUTHOR(S): Gabriel, S.; Wolter, Rheinhold
SOURCE: Ber. (1923), 56B, 2445-8
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
OTHER SOURCE(S): CASREACT 18:10950

L17 ANSWER 36 OF 49 CAPLUS COPYRIGHT 2005 ACS on STN
GI For diagram(s), see printed CA Issue.
AB cf. C. A. 10, 2724. The discovery that the quite readily accessible nitriles of the type ROCH2-C6H4CN can be smoothly reduced catalytically to the bases ROCH2C6H4CH2NH2 (C. A. 17, 2582) affords a new and important point of departure for the synthesis of aliphatic-aromatic compds. In the present paper is described the synthesis of o-homoxylylene bromide (I) by the following series of reactions: ROCH2C6H4CH2NH2 → ROCH2C6H4CH2OH → ROCH2C6H4CH2Br → ROCH2C6H4CH2CN → ROCH2C6H4CH2CO2H → ROCH2C6H4CH2CO2R' (II) → ROCH2C6H4CH2CH2OH → BrCH2C6H4CH2CH2Br (I). With R = Ph the above synthesis could not be carried out beyond the stage of the ester II, as in its reduction (with Na and alc.) the PhO group was also completely eliminated; even poorer results were obtained with R = Me but success was finally attained with R = Et. I has a characteristically greater tendency to ring closure than o-C6H4(CH2Br)2; the 2 and C atoms of the side chains can be brought together not only through another C atom to form tetralin derivs. or through a N atom to form tetrahydroisoquinoline derivs. but also through an O or S with formation of isochromans or thioisochromans. Hydrogenation of o-EtOCH2C6H4CN, b12 122° (which is obtained almost quant. and with extraordinary ease by heating NCC6H4CH2Br, instead of the chloride, with 1.1 atoms Na in alc.), in very concentrated decalin solution at 130° gives 40-52% of o-ethoxymethylbenzylamine (III), b12 130° (HCl salt, m. 152°; picrate, light yellow, m. 148°), and about 20% bis-[o-ethoxymethylbenzyl]-amine, reddish yellow, b12 237° (picrate, m. 93°); the Bz derivative, a thick oil, when heated 3 hrs. at 70° with somewhat more than its own weight of fuming HBr yields 70% of the compound (BrCH2C6H4CH2)2NBz, m. 124°, which, like PhCH2Br, reacts easily with Na, primary and sec. bases, NaCH(CO2Et)2, AcCHNaCO2Et, etc.). o-Phenoxymethylbenzyl alc., obtained in over 80% yield from the amine in AcOH with NaNO2, m. 50°, b16 216°; with concentrated HBr, even in the cold, the PhO group is replaced by Br almost as rapidly as the HO group, and the o-phenoxymethylbenzyl bromide, m. 54°, was obtained only by treating the alc. in cold CHCl3 with the calculated amount of
PBr3 in CHCl3 in small portions; yield, 55-60%. Cyanide, from the bromide with 2 mols. KCN in aqueous alc. on the H2O bath (yield, 90%), b17 220°, m. 78°, gives, after boiling 7 hrs. with 4 mols. of aqueous alc. KOH, more than 70% of o-phenoxymethylphenylacetic acid (IV), faintly yellow, m. 105°, which is quant. converted by boiling 4 hrs. in 10 parts alc. with 0.5 part concentrated H2SO4 into the Et ester, b16
225°; this with Na and alc. yields β-o-tolyl-ethyl alc., b15 120°, identical with the product obtained from o-MeC6H4CH2CO2Et. IV in the calculated amount of Na2CO3 gives almost quant. on concentration and cooling
the Na salt, 3.5 g. of which, heated 24 hrs. at 100° with 2 g. o-O2NC6H4CHO and 18 g. Ac2O, yields the compound PhOCH2C6H4C(CO2H):CHC6H4NO2, faintly yellowish, m. 152-3°; this is smoothly and quickly reduced by Fe(OH)2-NH4OH to the amino acid, m. 142°, yellow flocks becoming colorless on standing and recovering their yellow color in a desiccator, precipitated in colorless form from alc. by Et2O; treated in 5% KOH with NaNO2, then poured into an excess of cold 3% H2SO4 and shaken with Cu powder, the NH2 acid yields more than 50% 1-phenoxymethyl-10-carboxyphenanthrene (8-phenoxymethyl-phenanthrene-9-carboxylic acid), faintly yellowish, m. 201°. o-Ethoxymethylbenzyl alc., obtained practically quant. from the amine, b16 146°; bromide, prepared in 88% yield with PBr3, b16 135-7°; cyanide, b16 150°, hydrolyzed by alkalies to o-ethoxymethylphenylacetic acid (yield, 75%), b16 190°, whose Et ester, b17 156°; this with

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Na and alc. gives about 25% o-MeC6H4CH2CH2OH, b12 120°, and 35% β-o-ethoxymethylphenylethyl alc., b12 149-52°, which, heated 32 hrs. in a sealed tube at 100° with 4 parts fuming HBr, yields, besides about 25% of a substance (V) b10 about 100°, 60% of I, m. 53°, b10 168°, stable for weeks when protected from the light. The amt. of V, b12 90°, formed increases as the length of heating with HBr is diminished and after only 5 hrs. it may become the chief product of the reactions. It is isochroman, H2, C6hH4.CH2.CH2.O.CH2 as it is converted into I by heating with HBr and, conversely, is formed from I by warming with H2O or, better, with dil. K2CO3. The analogous thioisochroman, obtained in almost 40% yield from I boiled in aq. alc. with about 2 mols. K2S, b13 128-30°; HgCl2 compd., C9H10S.-HgCl2, m. 201°; methiodide, m. 123°. Di-Et ac-tetralin-β,β-decarboxylate, from I with 2 atoms Na and 1 mol. CH2(CO2Et)2 in alc., b13 180° free acid, m. 176° with stormy evolution of CO2 and formation of ac-tetralin-β-carboxylic acid, m. 97-8°. I heated several hrs. at 100° with 2 mols. NHMe2 in C6H6, shaken out with dil. HBr, made strongly alk., taken up in CHCl3 and treated with Et2O yields the extraordinarily hygroscopic N-dimethyltetrahydroisoquinolinium bromide, identified as the chloroplatinate, m. 230°. β-o-Tolylethyl bromide, from the alc. heated 6 hrs. at 120° with 3 parts fuming HBr, b16 112-5°; treated at 125-30° with 1 mol. Br it yields about 60% of a product, b16 140-80° which has approx. the compn. C9H10Br2 but which cannot be sepd., either by distn. or freezing out, into individual compds.; treated as above with NHMe2 it gives a quaternary Br compd. yielding the same chloroplatinate as above, the amt. of which indicates that only 25% of the 140-80° product consists of I; the remainder probably contains both Br atoms chiefly in the Et side chain. N- Phenyltetrahydroisoquinoline, obtained almost quant. from I with 3 mols. PhNH2, b16 198°, turns brown in the air rapidly; HCl salt, oily; picrate, yellow, m. 120°.

ACCESSION NUMBER: 1924:6065 CAPLUS
DOCUMENT NUMBER: 18:6065
ORIGINAL REFERENCE NO.: 18:829e-i,830a-f
TITLE: Syntheses in the aliphatic-aromatic series. XIV. Homo-o-xylylene bromide
AUTHOR(S): v. Braun, Julius; Zobel, Friedrich
SOURCE: Ber. (1923), 56B, 2142-52
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
OTHER SOURCE(S): CASREACT 18:6065

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GI For diagram(s), see printed CA Issue.
AB cf. C. A. 9, 2061. The formation of these anhydrides is characteristic of the o- and p-aminophenols, but not of the m-compds. 2,4-H2N(SO3H)C6H3OH (A) was prepared by the following steps: PhOH → p-HOC6H4SO3H → 2-O2N(HO)C6H3SO3Na → (A). When diazotized by the usual methods it yields the very soluble benzene-2-diazo-1-oxide-4-sulfonic acid (B), HO3SC6H3.O.N2, which, for purposes of isolation, is best prepared in the absence of non-volatile mineral substances, using purified N2O3 (C. A. 11, 1824). 1 g. finely powdered (A) was suspended in 5 cc. H2O and heated to boiling to dissolve most of the (A). After cooling in a freezing mixture 2 cc. N2O3 were added, giving a clear, intensely yellow solution from which (B) separated quant. as pale yellow crystals with 1 H2O of crystallization which is lost at 90° without decomposition of the compound or change of color. When quickly heated it blackens and decomps. violently 177°, but when kept at 115° it suddenly darkens and decomps. with gas evolution. The use of EtONO was unsatisfactory as a substitute for N2O3 but gave good results with "H acid." 4,2-H2N(HO3S)C6H3OH(C) was prepared by adding p-H2NC6H4OH to 3 parts H2SO4, heating 3 hrs. on the H2O bath, adding to H2O, and purifying by bone-blacking the Na salt. Phenol-4-diazonium sulfonate (D) was prepared from (C) by adding either EtONO or HCl and NaNO2 to a suspension in H2O at 0°. (D), dissolved in C5H5N, gave a yellow, crystalline salt which, however, lost all its C5H5N in vacuo over H2SO4. No crystalline product could be obtained from PhCH2NH2. (D), mixed with excess C5H10NH and placed in a desiccator over NaOH-CaO to exclude CO2, gave yellow piperidine benzene-4-diazo-1-oxide-2-sulfonate, purified by washing with PhH, turns brownish yellow on drying in a desiccator and then analyzes for C11H15O4N3S.2/3H2O, has an intense odor like acetamide. A suspension of (D) in cold H2O, treated with excess (PhCH2)2NH, gave dibenzylamine benzene-4-diazo-1-oxide-2-sulfonate, yellow crystals with 1 H2O of crystallization. PhNH2 also gives a yellow salt. All these salts, however, could be at least partially diazoamino compds., but since brucine is a tertiary amine, this objection could not apply to the brucine salt, from brucine HCl and (D) in H2O, followed by 1 equivalent of Na2CO3, bright yellow leaflets with 1 H2O; formulas (I) or (II) are assigned. Metallic salts were not isolated. At room temperature in the presence of excess NH3 (D) gives off its diazo N only very slowly, 88% being eliminated after 8 days, and very little tendency for azo compound formation being shown. m-H2NC6H4OH was sulfonated as in the case of the p-compound, the acid purified by recrystn. from H2O, and diazotized in the form of a finely divided suspension obtained by acidifying a solution of the Na salt with HCl. The resulting phenol-3-diazonium-4-sulfonate (E), HOC6H3.SO2.O.N2, forms a yellowish white precipitate which decomps. at 86° with effervescence, contains H2O of crystallization, and loses N even at room temperature. An attempt to prepare the brucine salt failed, only a few orange-colored crystals being obtained. The orange color is due to the very soluble dye (III), which forms when (E) is treated with excess NH3, only 0.5 the diazo N being evolved.

ACCESSION NUMBER: 1917:11980 CAPLUS
DOCUMENT NUMBER: 11:11980
ORIGINAL REFERENCE NO.: 11:2458e-i,2459a-e
TITLE: Constitution of internal diazo-oxides (diazophenols). II
AUTHOR(S): Morgan, Gilbert T.; Tomlins, Henry P.
CORPORATE SOURCE: Finsbury Techn. Coll., London

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SOURCE: Journal of the Chemical Society, Abstracts (1917), 111, 497-506
CODEN: JCSAAZ; ISSN: 0590-9791
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

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GI For diagram(s), see printed CA Issue.
AB cf. C. A., 6, 1139. 2,4-Dimethylbenzylhydrazine, from the monohydrochloride with CaO, b13 136-7°. Extremely unstable. Dihydrochloride, from the monohydrochloride and dry HCl. White powder, m. 164°. Unstable. Sulfate, small crystals, m. 163°. Oxalate, colorless crystals, m. 192°. Picrate, yellow needles, m. 148°. From the monohydrochloride, the following compds. were obtained: By b. with dilute HCl, 2,4-dimethylbenzyl chloride, colorless oil, b19 103-4°. With Ac2O, diacetyl-2,4-dimethylbenzylhydrazine, colorless plates, m. 129°. With KOCN, 2,4-dimethylbenzylsemicarbazide, columnar prisms, m. 162°. With PhNCS, 2,4-dimethylbenzylphenylthiosemicarbozide, colorless prisms, m. 138-5°. With AcONa and tartaric acid, α-2,4-dimethylbenzylhydrazonepropionic acid, pasty consistency. With NaNO2, nitroso-2,4-dimethylbenzylhydrazine, colorless plates, m. 60.5°, which condenses with 2,4-Me2C4H3CHO to form Me2C4H3CH2N(NO)N : CHC6H3Me2. 2,4-Dimethylbenzyl azide, by heating with 10% H2SO4 at 80°, colorless oil, b15 114°. Ethyl β-2,4-dimethylbenzylaminocrotonate, from 2,4-Me2C4H3CH2NHNH2 and AcCH2CO2Et, colorless plates, m. 85°. N-2,4-Dimethylbenzyl-3-phenyl-5-pyrozelone, from the hydrazine and BzCH2CO2Et, colorless needles, m. 162°. With NaNO2 and AcOH, the pyrazolone gave N-2,4-dimethylbenzyl-3-phenyl-4-isonitroso-5-pyrazone (I), fine red needles, m. 128° (decompose). N-2,4-Dimethylbenzyl-2-methyl-3-phenyl-5-pyrazolone, from 2,4-dimethylbenzylphenylpyrazolone, in MeOH, and MeI at 120°, brown oil. Gave an intense red color with FeCl3, and green color with NaNO2 in the presence of a trace of acid. N-2,4-Dimethylbenzyl-3-methylpyridazione (II), from 2,4-Me2C6H3CH2NHNH2.HCl, AcONa and levulinic acid, large crystals, m. 79.5°. The following derivs. of (2,4,5-Me3C6H2CH2)2NNH2 were prepared: Sulfate, white needles, m. 151°. Nitrate, small plates, m. 118°. Chloroplatinate, dark red precipitate m. 95°. Acetone-2,4,5-trimethyldibenzylhydrazone, needles, m. 132°. Heated with Me2CHCHO on the H2O bath for 4 hrs., it gave isobutyraldehyde-2,4,5-trimethyldibenzylhydrazone, small needles, m. 112°; with Ac2O, diacetyl-2,4,5-trimethyldibenzylhydrazine, white needles, m. 126°; with BzCl, the monobenzoyl derivative, needles, m. 129°; with EtI, 2,4,5-trimethyldibenzylethylazonium iodide, white needles, m. 160°; with HgO (yellow), 2,4,5-trimethyldibenzyltetrazone, small plates. When the (Me3C6H2CH3)2NNH2.HCl was treated with KOCN, 2,4,.5-trimethyldibenzylsemicarbazide was formed. Small white plates, m. 173°. p-Isopropylbenzyl-p-isopropylbenzalhydrazone, from (C3H7C6H4CH : N)3 and Na-Hg, small yellowish green prisms, m. 75° (decompose). Very unstable. Benzoyl derivative, crystalline powder, m. 78°. Nitroso derivative, bright yellow fibrous needles, m. 59°. sym.-p-Isopropyldibenzylhydrazine hydrochloride, obtained by reduction of (p-C3H7C8H4C: N)2 with Na-Hg, hexagonal tablets, m. 217° (decompose); 50% yield. The free hydrazine was not obtained in crystalline form; it was identified as the hydro-chloride. Diacetyl derivative, rhombic prisms, m. 71°. Dinitroso derivative, yellow needles, m. 59°. Warmed with absolute alc., the dinitroso derivative yielded C3H7C6H4CH2N(NO)N: : CHC8H4CZH7, while (p-C3H7C8H4CH : N)2 was formed when it was b. with absolute alc. p-Isopropyldibenzal-p-isopropyldibenzylhydrotetrazone, by b. [C3H7C6H4CHZN(NO)]3 in alc. solution, m. 165-9°. p-Isopropylbenzylhydrazine hydrochloride, from C3H7C6H4CH2NHN : CHCH6H4C3H7 and dilute HCl, needles, sinter 143°, m. 199°. The free base, crystalline, m. 46°. Nitroso derivative, fibrous crystals, m. 63°. p-Isopropylbenzyl azide, from the above nitroso derivative warmed with 10% H2SO4, b23 118°. Stable toward

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alks. (m-ClC8H4CH2)2NH was prepared by reducing (m-ClC6H4CH : N)2, with Zn dust and AcOH. m-Chlorodibenzylamine nitrite, white glistening plates, m. 133°; when heated for 5-6 hrs. with abs. alc. on the H2O bath it gave m-chlorodibenzylnitrosoamine, yellow needles, m. 53°, which yielded, on reduction with Zn dust and AcOH, benzylidene-m-chlorodibenzylhydrazone, yellow needles, m. 66°. With HCl and steam the hydrazone gave asym.-m-chlorodibenzylhydrazine hydrochloride, white plates, m. 200° (decomp.). sym.-m-Chlorodibenzylhydrazine hydrochloride, by reduction of (m-ClC6H4CH: N)2, with Na-Hg, light yellow needles, m. 191°. The free base, white needles, m. 43°, unstable. Dibenzoyl derivative, from the hydrochloride and BzCl, m. 88°. Stable in air. Diacetyl derivative, from the hydrochloride and Ac2O, colorless crystals, m. 73°. Dinitroso derivative, yellow crystals, m. 48° (decomp.). Warmed with alc., the nitroso deriv. gave nitroso-m-chlorobenzyl-m-chlorobenzalhydrazone, yellow needles, m. 98°. The hydrazone on hydrolysis with HCl yielded m-chlorobenzylhydrazine hydrochloride, needles, m. 134°. sym.-o-Hydroxydibenzylhydrazine, (HOC8H4CH2NH)2, obtained by reducing the aldazine with Na-Hg in alc., white plates from alc. by addition of H2O, m. 117°, yield 72%; it reduces hot alk. AgNO3 and is unaffected by b. dil. HCl. From the hydrazine were obtained the following compds.: Dihydrochloride, fine needles, m. 143°. With Ac2O it reacts spontaneously yielding the diacetyl derivative (HOC6H4CH3NAc)2, white plates from dil. alc., m. 178-9°. With Ac2O on the H2O bath for 3-4 hrs., the diacetyl diacetate was formed, cryst., insol. in alc. With NaNO3 and AcOH, nitroso derivative, yellowish brown crystals, m. 90° (decomp.), unstable in air, and when b. with EtOH yields o-hydroxybenzol-o-hydroxybenzylnitrosohydrazone, needles, m. 145°, decomposes with b. H2O. (m-HOC6H4CH: N)2 was obtained from m-HOC6H4CHO and N2H4.H2SO4 yellow needles, m. 205°. Sol. in NaOH with deep yellow color. When reduced with Na-Hg it yields m-hydroxydibenzylhydrazine, light yellow needles, m. 183°, stable. Yield, 79%. It reduces hot alk. AgNO3. Dihydrochloride, white cryst. ppt., m. 154°. The hydrazine did not yield the expected hydrotetrazone when treated with HgO, but gave m-HOC6H4CHO; with Ac2O it formed m-hydroxydibenzyldiacetylhydrazine, white crystals from dil. alc., m. 209°. Diacetyl diacetate, crystals from alc., m. 132°. m-Hydroxybenzal-m-hydroxybenzylnitrosohydrazone, white needles from H2O, decomp. 112-4°, gives Lieberman's reaction with PhOH. o-Ethoxydibenzylamine, (EtOC6H4CH2)2NH, prepared by reducing (o-EtOC6H4CH : N)2 with Zn dust and AcOH, yellow oil, b20 180°, non-volatil with steam. Chloroplatinate, red ppt., insol. in alc. and H2O. By reducing the corresponding methoxy deriv. o-methoxydibenzylamine was obtained, b30 200°. No definit compd. was obtained by reducing the benzoxy deriv. o-Methoxybenzyl-o-methoxybenzalhydrazone, by reducing (o-MeOC6H4CH : N)2 with Na-Hg, white needles from alc. m. 76°, unstable, yield 69%. With Ac2O it yields acetyl-o-methoxybenzyl-o-methoxybenzalhydrazone, prisms from hot alc., m. 101°, stable. Benzoyl derivative, prisms from alc., m. 170°. Nitroso derivative, bright yellow needles, m. 91°. Reduction of (o-MeOC6H4CH : N)2 by Na-Hg yielded o-methoxydibenzylhydrazine hydrochloride, long white needles, m. 154°. Turns yellow in air. Diacetyl derivative, white crystals from alc., m. 133-4°. o-Methoxybenzylhydrazine hydrochloride, obtained by hydrolyzing MeOC6H4CH2NHN : CHC6H4OMe with dil. HCl, m. 123-4°. The free base was obtained by decomp. the hydrochloride with NaOH. Colorless liquid, b14 145-9°. From this base were obtained the following compds.: with AcCH2CO2Et, 1-o-methoxybenzyl-3-methyl-5-Pyrazolone, clusters of red needles, m. 82-4°, gives yellow ppt. with HNO2. With tartaric add, α-o-

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methoxybenzylhydrazonepropionic acid, crystals from alc., m. 107.5°. With KOCN, o-methoxybenzylsemicarbazide, white crystals, m. 214-5°. Nitroso-o-methoxybenzylhydrazine, fine white needles from H2O, m. 65°, quant. yield. This nitroso deriv. condenses with o-MeOC6H4CHO to form MeOC6H4CH2N(NO)N : CHC6H4OMe, and when hydrolyzed by 10% H2SO4 it gave o-methoxybenzyl azide, colorless liquid, b14 118°. The azide was unaffected by b. for 4 hrs. with 30% NaOH, but decomp. when b. for 10 hrs. with 30% H2SO4. When reduced with Na-Hg it gave o-hydroxybenzyl-o-methoxybenzalhydrazone, white cryst. powder, insol. in all ordinary reagents, turns yellow at 115°, m. 153-7°. It forms a yellow insol. nitroso derivative. sym.-m-Methoxydibenzylhydrazine, hydrochloride, by reducing (m-MeOC6H4CH : N)2 with Na-Hg and satg. with dry HCl, white needles, m. 115°. Yield, 60%. Free base, light yellow oil. The hydrochloride gave with NaNO2, nitroso-m-methoxybenzyl-m-methoxybenzalhydrazone, yellow needles, m. 80°. m-Methoxybenzylhydrazine hydrochloride, by reducing (m-MeOC6H4CH : N)2 with Na-Hg, white triclinic prisms, m. 123°, becomes yellow in air and reduces cold alk. AgNO3. Yield, 35%. The free base, b19 158-68°, loses N both in air and in vacuo. From the hydrochloride were obtained the following compds.: dibenzyl-m-methoxybenzylydrazine, white needles from alc., m. 128°. With tartaric acid, α-m-methoxybenzylhydrazonepropionic acid, rhombic plates, m. 99°. Nitroso-m-methoxybenzylhydrazine, small needles from alc., m. 45-7°. It condenses with m-MeOC6H4CHO to form MeOC6H4CHN(NO)N : CHC6H4OMe, and yields m-methoxybenzyl azide by distn. with 10% H2SO4, colorless oil, b28 134°. It decomp. by b. with 30% H2SO4. When (m-MeOC3H4CH : N)2 was reduced in acid soln. with Zn dust, m-methoxydibenzylamine hydrochloride was formed. White leaflets, m. 141°; 85% yield. The free amine, colorless liquid, b13 225°. Nitrate, needles, m. 128°. Picrate, yellow tablets, m. 124°. Nitrite, white needles from alc., m. 104°. The primary amine, m-methoxybenzylamine. hydrochloride, was obtained by reducing the benzaldazine in very dil. alc. soln. with Zn dust and AcOH, needles, m. 160°. Reduction of (p-MeOC6H4CH : N)2 in alk. soln. (Na-Hg) yielded p-methoxybenzyl-p-methoxybenzalhydrazone, white plates, m. 143°. Nitroso derivatives bright yellow leaflets, decomp. 106°. Acetyl derivative from Ac2O, white needles from alc., m. 87°. Benzoyl derivative, white needles, m. 111-2°. Picrate, yellow fibrous needles, m. 90°. The hydrazone yielded on b. with conc. HCl, p-methoxybenzylhydrazine hydrochloride, yellow crystals, decomp. 194-5°; 69-74% yield. Free base, b14.5 170-5°. The following compds. were obtained from the hydrochloride: With tartaric acid, α-p-methoxybenzylhydrazonepropion ic acid, white needles from dil. alc., m. 123-4°. With BzCl, dibenzoyl-p-methoxybenzylhydrazine, colorless prisms, m. 149°. With NaNO2 and AcOH, nitroso-p-methoxybenzylhydrazine, large white tablets, m. 91°; 64% yield. The nitroso deriv. condenses with p-MeOC4H4CHO to form MeOC6H4CH2N(NO)N : C6HC6H4CHO, and forms p-methoxybenzyl azide when warmed with 10% H2SO4, colorless oil, b14 126°. It is unaffected both by b. with NaOH and on distn. with steam, but decomp. when b. for 4 hrs. with 30% H2SO4. sym.-p-Methoxydibenzylhydrazine hydrochloride, obtained by reducing (p-MeOC6H4CH : N)2 with Na-Hg, colorless plates, m. 236-7° (decomp.). Free base, plates, m. 71°. Nitrite, needles from alc., m. 92° (decomp.). Diacetyl-p-methoxydibenzylhydrazone, from the hydrazine and Ac2O, fine white plates from dil. alc., m. 113°, (p-MeOC6H4CH : N)3 on reduction in acid soln. gave (p-MeOC6H4CH2)2NH.HCl. Nitrite of the amine, prisms, m. 147°. Piperonaldazine monohydrochloride, (CH2O2 : C6H3CH : N)2.HCl, was obtained from the aldazine and conc. HCl, dark

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yellow plates, m. 207°. Dihydrochloride, obtained by satg. the aldazine in CHCl3 with dry HCl, yellow ppt., m. 213°. Unstable. Sulfate, deep yellow ppt., m. 221°. Tetrabromido, red powder, decomp. 185°. Dihydrobromide, from the tetrabromide and Me2CO, yellow powder. Monohydrobromide, cryst. powder, m. 216°. The aldazine on reduction with Na-Hg gave piperonylpiperonalhydrazone, white plates and needles, sinter 109°, decomp. 116°. Turns yellow in air. From the hydrazone were obtained the following compds.; nitroso derivative, yellow needles, decomp. 145°. Acetyl derivative, clusters of plates, m. 146°. Benzoyl derivative, white needles from alc., m. 125°. Piperonylhydrazine hydrochloride, by hydrolysis with H2SO4, fine white needles, m. 173.5°, stable in air when pure. It reduces warm Fehling soln. and cold alk. AgNO3. From the hydrazine hydrochloride were obtained the following compds.: With KOCN, piperonylsemicarbazide, CH2O2 : C6H3CH2N(NH2)CONH2 white needles, m. 175°. With KOH and PhNCS in alc. soln., piperonylphenylthiosemicarbazide, needles from alc., m. 153.5°. With HN3, nitrosopiperonylhydrazine, needles, m. 91°. The nitroso deriv. yielded on hydrolysis with dil. H2SO4 (1 : 10) piperonyl azide, CH2O2 : C6H3CH2N3, b13 142°. Stable toward b. alk. but decomp. with 50% H2SO4. Piperonylhydrazine, from its hydrochloride, yellow oil, b14 175-80°. Unstable in air. With tartaric acid it gives α-piperonylhydrazonepropionic acid, plates, m. 143°, and with AcCH2CO2Et, 1-piperonyl-3-methyl-5-pyrazolone, small needles, m. 155°; 77% yield; acid to litmus, gives yellowish red color with FeCl3, and forms a silver salt with AgNO3. The pyrazolone with NaNO2 and AcOH yielded 1-piperonyl-3-methyl-4-isonitroso-5-pyrazolone, bright yellow needles, m. 161°; 74% yield. 1-Piperonyl-3-phenyl-5-pyrazolone, from piperonylhydrazine and BzCH2CO2Et. Cryst. powder, m. 144.5°; 90% yield. 1-Piperonyl-3-phenyl-4-isonitroso-5-pyrazolone, made like 3-methyl compd., red powder, m. 162°. 1-Piperonyl-3-methylpyridazinone, from piperonylhydrazine and levulinic ester, long needles from H2O, m. 101°. Piperonaldazine on reduction with Na-Hg, and acidification with HCl, gave sym.-dipiperonylhydrazine hydrochloride, m. 223°. Free base, yellow plates, m. 88°. It reduces alk. AgNO3, but not Fehling soln. Unstable in air. With Ac2O it forms diacetyldipiperonylhydrazine, plates, m. 138°. With BzCl, diberizoyl derivative, small yellow plates, m. 98°. With NaNO3 and HCl, dinitroso derivative, white plates, m. 95°. o-Chlorobenzaldazine tetrabromide, made by the action of Br in CCl4 upon (o-ClC6H4CH : N)2 in CCl4, m. 172-5°. α-Naphthaldazine tetrabromide, from the aldazine and Br in CCl4. Red powder, m. 170-2°. Decomp. by cold alc. and Me2CO.

ACCESSION NUMBER: 1912:17249 CAPLUS
DOCUMENT NUMBER: 6:17249
ORIGINAL REFERENCE NO.: 6:2396f-i,2397a-i,2398a-i,2399a-i,2400a
TITLE: Reduction of Aromatic Aldazines
AUTHOR(S): Curtius, Theodor
CORPORATE SOURCE: Univ. Heidelberg
SOURCE: Journal fuer Praktische Chemie (Leipzig) (1912), 85, 137-88,393-484
CODEN: JPCEAO; ISSN: 0021-8383
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

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AB cf. preceding abstract p-Methoxybenzyl chloride, from the alc. and dry HCl, b15, 116-20°, d0 1.072. Bromide, b6 129°, d19 1.395. Either the chloride or bromide, mixed in a sealed tube with 20% MeNH2 in alc., gives p-methoxybenzylmethylamine, b14 121°, d0 1.025°; hydrochloride, m. 166°; hydroiodide, m. 145°, heated with concentrate HI, gives p-hydroxybenzylmethylamine hydroiodide, m. 149-50°; hydrochloride, m. 188-90°. In the prepare of MeOC6H4CH2NHMe is also formed di-p-methoxybenzylmethylamine, b13 223-5°, d0 1.0794. Di-p-hydroxybenzylmethylamine hydrochloride, m. 197-9°. With MeZNH instead of MeNH2 is obtained p-methoxybenzyldimethylamine, b16 110-1°, d0 0.9878, d15 0.976; hydrochloride, m. 157°; hydroiodide, m. 145°; methiodide, m. 158°. Ac2O decompose the base into MeOC6H4CH2OAc and AcNMe2. p-Hydroxybenzyldimethylamine, m. 112°, alkaline to litmus and phenolphthalein, does not appreciably color aqueous FeCl3, reduces NH3-AgNO3, Millon's reagent and HI, decompose by Ac2O into AcOC6H4CH2OAc and AcNMe2. The methiodide m. 158° (above), heated with concentrate HI, gives p-hydroxybenzyltrimethylammonium iodide, m. 191°; chloride, m. 98°.

ACCESSION NUMBER: 1911:22223 CAPLUS
DOCUMENT NUMBER: 5:22223
ORIGINAL REFERENCE NO.: 5:3803i,3804a-c
TITLE: Monomethyl- and Dimethyl-p-hydroxybenzylamine
AUTHOR(S): Tiffeneau, M.
SOURCE: Bull. soc. chim. (1911), 9, 825-8
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

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GI For diagram(s), see printed CA Issue.
AB The method of preparing phenylcamphoformeneaminecarboxylic acid, formula I, was improved. (Am. Chemical J., 21, 250). On adding 4 ats. Br in CHCl3 to (I) in CHCl3, 3,4-dibromoaniline hydrobromide, and camphoroxalic acid resulted. CrO3 does not attack (I) at room temperature but in moist acetone KMnO4 oxdizes (I), yielding camphorquinone. PCl3 or PCl5 with (I) produces a tarry mass from which only camphoroxalic acid could be isolated. Me2SO4 and KOH on (I) yield the methyl ester, yellow crystals from MeOH, m. 127°. The conditions were varied widely but neither the NHPh not the : COH group appeared to be attacked. Me2SO4 and Na2CO3 at 100° had no action on phenylcamphoformeneamine. Camphoroxalic acid (II) yields with Me2SO4 and KOH the methyl ester, which with Me2SO4 and Na2CO3 at 150-80° yields an oil, probably methyl methoxycamphoroxalate. HNO2 from NaNO2 or amyl nitrite failed to react with (I), (II) or the ethyl ester of (II). Thiosemicarbazine and (II) react rapidly in boiling, slowly in cool alc., to form thiosemicarbazylcamphoformeneaminecarboxylic acid, (III), which exists in 2 forms, (a) white flakes from C6H6, m. 148.9° almost insol. in C6H6, (b) white powder, precipitated from alkaline solution by HCl, m. 120-5°, readily soluble in C6H6, being deposited from it as (a), hence probably an unstable hydrate of (a). When fused (III) gives a resin and a small quantity of a compound, m. about 170°. Ethyl ester of (III) white crystals from C6H6, m. 150-1°. On dissolving (III) in Ac2O, thiosemicarbazylcamphoformeneaminecarboxylacetimide, (IV) is formed rapidly at 100°, slowly at room temperature, bright red crystals from glacial AcOH, m. 181-2°, dissolves in warm KOH, forming salt of (III). 1 g. of (III) was mixed with 1.5 cc. Al2O. The addition of 3 drops concentrate H2SO4 generated heat and formed a clear solution After 15-20
min. the solution was poured into H2O, camphylpyrazolecarboxylic acid m. 261-2° was isolated, (Am. Chemical J., 36, 259); the solution contained HCNS. H2SO4 on (III) formed only a tarry material. The replacement of CO by CS in these condensation products reduces the tendency to form cyclic derivs. Camphoroxalic acid and 1,3,4-xylidine (2 mols.) warmed together in C6H6, give 1,3,4-xylidine 1,3,4-xylidylcamphoformeneaminecarboxylate, (V) brown crystals from ligroin, m. 93-4°. 1,3,4-Xylidylcamphoformeneaminecarboxylic acid, by the action of KOH on (V), or by warming (II) and the amine in C6H6, till a drop of the solution gave no color with alc. FeCl3, yellow crystals from ligroin, m. 117-8°. p-Clorophenylcamphoformeneaminecarboxylic acid, yellow needles from C6H6, m. 182-3°. When an intimate mixture of (II) and p-chloroaniline is heated, it m. 65-70°, evolves H2O about 110° and then solidifies, m. again about 155° and evolves CO2; a 61% yield of p-chlorophenylcamphoformeneamine, (VI) was obtained, white crystals, from acetone and ligroin m. 194.5°, is unchanged by boiling KOH or HCl. Camphoroxalic acid and the amine (1 or 2 mols.) in warm C6H6 yield **dibenzylamine** carboxylate, white crystals from C6H6, m. 135-6°. Heated with 2 mols. PhNH2 for 5 hrs. at 100° in a sealed tube, it yields **dibenzylamine** phenylcamphoformeneaminecarboxylate, white crystals from C6H6, m. 185°. A 75% yield of dibenzylcamphoformeneamine (Am. Chemical, J., 39, 117) was obtained by heating 1 mol. of camphoroxalic acid and 1 or 2 mols. of the amine at 135-40° for 30 min. m-Aminobenzoic acid (1 or 2 mols.) and (II) in alc. solution yield m-carboxyphenylcamphoformeneamine
carboxylic acid, white crystals from alc., m. 136-7°, with alc. FeCl3 gives no color, but is hydrolyzed by H2O or 50% alc. When fused this acid evolves CO2 and forms m-carboxyphenylcamphoformeneamine, long, yellow needles from C6H6, m. 116-7°. On warming C6H6 solns.

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of (II) and benzidine (1 mol.) the inner ammonium salt (VII) was formed, yellow crystals from C6H6, m. about 208° depending on rate of heating. (Am. Chem. J., 34, 231; 36, 229). The fact that it dissolves only slowly in boiling KOH, indicates the structure given, rather than that for benzidylcamphoformeneaminecarboxylic acid, although it is repptd. from alkaline soln. by HCl. Benzidylcamphoformeneamine, m. 317-8°, is obtained by the fusion of (VII), or better by heating a mixture of (VII) in 5 parts PhNO2 at 150-5° for 15 min. On heating camphylamine and (II) at 150-5°, a white crystallin sublimate, m. 105° was formed. The results support the formulas similar to (I), (VI), etc., previously assigned to the condensation compds. (cf. C. A., 2, 1009, 1129).

ACCESSION NUMBER: 1911:1726 CAPLUS
DOCUMENT NUMBER: 5:1726
ORIGINAL REFERENCE NO.: 5:282i,283a-i,284a-c
TITLE: Derivatives of Camphoroxalic Acid. XIII
AUTHOR(S): Tingle, J. Bishop; Bates, S. J.
CORPORATE SOURCE: McMaster Univ., Toronto
SOURCE: Journal of the American Chemical Society (1911), 32, 1499-1517
CODEN: JACSAT; ISSN: 0002-7863
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L17 ANSWER 41 OF 49 CAPLUS COPYRIGHT 2005 ACS on STN
GI For diagram(s), see printed CA Issue.
AB Tert. butyldihydroisoindole, formula (I) below, is prepared by boiling o-xylylene bromide, tert. butylamine and KOH with alc.; lustrous plates, m. 42°; b13 125-30°. Methiodide, from MeI and MeOH; colorless crystals from alc., m. 221°. p-Acetophenyldihydroisoindole (II) from p-aminoacetophenone and o-xylylene bromide; lustrous plates from acetone, alc., glacial AcOH or pyridine, m. 197°. Benzal derivative, C8H8 : NC6H4COCH : CHPh, yellow, silky lustrous plates from alc., m. 202°. Cinnamylidene-p-acetophenyldihydroisoindole, C8H8 : NC6H4COCH : CHCH : CHPh, prepared in a similar manner to the preceding compound; slender, orange-colored needles from acetone, m. 187°. It gives a blood-red color with concentrate H2SO4. p-Nitrobenzal-p-acetophenyldihydroisoindole, C8H8 : NC6H4COCH : CHCH6NO2, light yellow, crystallin powder from pyridine, m. 238°. It gives a purple-red color with concentrate H2SO4 and an intense orange shade with concentrate HCl or HNO3. p-Dimethylaminobenzal-p-
acetophenyldihydroisoindole, C8H8 : NC6H4COCH : CHC6H4NMe2, from p-dimethylaminobenzaldehyde; golden yellow plates from pyridine, m. 196°. The following derivs. of phenyldihydroisoindole have been prepared from the compds. mentioned. Methiodide, C8H8 : NPhMeI, from MeI and MeOH; colorless plates from alc. + Et2O, m. 177°. Bisxylyleneaminodiphenylmethane, (C8H8 : NC6H4)2CH2., from HCHO, at 125°; aggregates of slender needles from pyridine, m. 308-9°. With oxidizing agents it gives a deep blue dye. Bisxylyleneaminotriphenylmethane, (C8H8: NC6H4)2CHPh, from BzH, in presence of fuming HCl, slender, interlaced, snow-white needles from pyridine + alc., m. 265°. It becomes blue and light yellow when warmed with acids and alkalies, resp. Bisxylyleneaminodimethylaminotriphenylmethane, (C8H8 : NC6H4)2CHC6H4NMe2, from p-dimethylaminobenzaldehyde; colorless, stellate needles from pyridine, m. 185°. Bisxylylemaminodiphenylcinnamenylmethane, (C8H8 : NC6H4)2CHCH : CHPh, from cinnamic aldehyde; yellow, crystallin powder from pyridine + alc., not m. 300°. m-Tolyldihydroisoindole, C8H8 : NC6H4Me[N : Me = 1 : 3] and HCHO form bisxylyleneaminodi-m-tolylmethane, (C8H8 : NC6H3Me)2CH2; colorless needles from pyridine, m. 255°. o-Xylylenedi-o-toluidine and HCHO, in pres. of concentrate HCl, give methylenedi-o-tolyl-o-xylylenediamine (III); colorless, lustrous scales from pyridine + alc., m. 139°. It does not react with BzCl or benzenesulphonyl chloride. Benzaldi-o-tolyl-o-xylylenediamine, from BzH, in a similar manner to the preceding compound; crystallin powder from CHCl3 + Et2O, m. 180°. At 200° xylylenepiperidinium bromide and MeNH2 give Pentamethylenemethylxylylenediamine (IV); water-clear oil with an odor of piperidine, b15, 160-5°. Benzenesulphonyl derivative, colorless crystals from alc., m. 87°. A little N-methyldihydroisoindole, C8H8 : NMe, is formed together with (IV). At 200° PhNH2 and xylylenepiperidinium bromide give piperidine and phenyldihydroisoindole. Amylenedihydroisoindole, C8H8 : NCH2CH2CH2CH : CH2, is produced by treating xylylenepiperidinium bromide with H2O and Ag2O and distilling the resulting alkaline liquid; liquid, b12 140-50°. It immediately decolorizes KMnO4, in presence of dilute H2SO4. Salts and methiodide, oily. o-Xylylenepentamethylenexylylenediamine, (C8H8 : NCH2)2C6H4, is obtained by heating p-dixylyleneammonium bromide, piperidine and H2O, at 200°; oil, b15 240-5°. o-Xylylenetetrahydroquinolinium iodide, C8H8 : NI : C9H10, is prepared by the interaction of o-xylylene bromide and tetrahydroquinoline, the oily product being treated with Ag2O followed by III; colorless needles from H2O, m. 238°. Picrate, yellow needles from H2O, m. 165°. o-Xylylenedibenzylammonium bromide, C8H8 : NBr(CH2Ph)2, from o-xylylene bromide and

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dibenzylamine; snow-white plates from H2O, m. 188°. At 200°, NH3 converts it into dibenzylxylylenediamine, C6H4(CH2NHC7H7)2; oil. Hydrochloride, colorless plates from alc. + Et2O, m. 251°. o-Xylylenediisoamylaimmonium iodide, C8H8 : NI(C5H11)2, is obtained from o-xylylene bromide and diisoamylamine, the product being treated with KI; white crystals from H2O, m. 139°. Bromide, hygroscopic. With NH3, at 200°, it is converted into diisoamylxylylenediamine, C6H4(CH2NHC5H11)2; colorless oil, b12 210°. Dibenzylpiperidinium bromide, C5H10 : NBr(CH2Ph)2, is prepared from 1,5-dibromopentane and **dibenzylamine**; white plates from alc. + Et2O, m. 253°. With NH3, at 200°, it is decomp. into **dibenzylamine**, benzylpiperidine and benzylamine. Dipropyla mine and o-xylylene bromide form o-xylylenedipropylammonium bromide, C8H8 : NBrPr2; colorless plates from acetone, m. 107°. At 200°, NH3 converts it into PrBr and N-propyldihydroisoindole, C8H8 : NPr; almost colorless oil, b. 230-40°. Methiodide, white, crystallin powder from alc. + Et2O, m. 150°. Chloroplatinate, reddish yellow, granular, crystallin powder from H2O, m. 192°, previously darkening.

ACCESSION NUMBER: 1910:17952 CAPLUS
DOCUMENT NUMBER: 4:17952
ORIGINAL REFERENCE NO.: 4:3218h-i,3219a-i,3220a-b
TITLE: Syntheses with o-Xylylene Bromide
AUTHOR(S): Scholtz, M.; Wolfrum, R.
CORPORATE SOURCE: Chem. Inst.;Univ. Greifswald
SOURCE: Ber. (1910), 43, 2304-18
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L17 ANSWER 42 OF 49 CAPLUS COPYRIGHT 2005 ACS on STN
GI For diagram(s), see printed CA Issue.
AB Potassium 3,5-dinitro-4-anilino-4-methoxyquinolnitrolate, is prepared from KOMe and picrylaniline; like the other salts of this series it is represented by formula (I) below, in which R indicates the alc. alkyl and M the metal. In common with a number of similar compds., which are described in this abstract, it is explosive and is best analyzed by moistening with alc. in a Pt crucible, then covering with dilute H2SO4and heating on the water bath during 1 hr. With excess of KOMe it gives a red product and with 50% aqueous KOH it becomes yellow. Potassium 3,5-dinitro-4-anilino-4-ethoxyquinolnitrolate, from alc. KOH in C6H6; bundles of dark brown needles with a bronze luster, m. about 115° (decompose); at a higher temperature it explodes. Yield, 85% of the picrylaniline. Dipotassium 1-anilino-1,3-diethyl-6-nitrocyclohexene-2,4-dinitrolate (II), from excess of alc. KOH, or KOEt in C6H6; small, dark red crystals with a metallic reflex, darkens about 120°, not m. 240°. Tripotassium 1-anilino-1,3,5-tripropoxycyclohexane-2,4,6-trinitrolate (III), from excess of alc. KOH; yellow, highly hygroscopic, crystalline powder; with alc. it gives (II). Potassium 3,5-dinitro-4-anilino-4-propoxyquinolnitrolate (see I) from KOH in PrOH; black plates with a blue luster. Tripotassium 1-anilino-1,3,5-tripropoxycyclohexane-2,4,6-trinitrolate (see III), from the mono-K salt; light brick-red powder. The only derivative of isobutyl alc. which could be obtained was tripotassium 1-anilino-1,3,5-triisobutoxycyclohexane-2,4,6-trinitrolate (see III); orange-yellow solid. Picrylmethylaniline, MeOH and KOH give a dark red solution, but no solid salt could be isolated. With alc. tripotassium. 1-methylanilino-1,3,5-triethoxycyclehexane-2,4,6-trinitrolate (see II) is produced, contrary to the statement of Sudborough and Picton; brick-red, amorphous, unstable powder. Dipotassium 1-anilino-1,3-dipropoxy-6-nitrocyclohexene-2,4-dinitrolate (see II) was the only compound which could be obtained from PrOH; brownish red powder. Isobutyl alc. gives, apparently, a mixture of di- and tyipotassium salts: dark red and amorphous. In order to avoid repetition of the names, the remaining compds. described in this abstract will be indicated by giving the number of mols. of the resp. K alcoholates which added themselves to the nitro compds. employed, the type formula will also be indicated. Picryl-β-naphthylamine, KOMe (see I); aggregates of black needles, m. about 173°. KOEt, aggregates of long, black, lustrous needles, m. 1680. A red and a yellow derivative were also prepared, probably they are the di- and tri- K compds. (see II and III). Isobutyl alc. and KOH gives a substance, C16H9O6N4KC4H9OK; light red and amorphous. Picryl-α-naphthylarnine, KOH and MeOM or EtOH form potassium picryl-α-naphthylamine, C16H9O7N4K; black crystals with a blue, metallic luster, m. above 230°. It is attacked only slowly by H2O. In presence of C6H6, excess of alc. KOH causes the deposition of a yellow unstable salt, probably a tri-K derivative (see III). When boiled for a short
time in alc. picryl chloride and methyl-α-naphthylamine form an additive compound (O2N)3C6H2ClC10H7NHMe; long, dark red, silky lustrous, interlaced needles, m. 94°. K picryl-α-naphthylamine when treated with a Ag salt at the ordinary temperature gives an oxidation product,
C16H10O7N4; brownish orange or brick-red, slender, interlaced needles from C6H6, m. 296-7°. When rubbed it becomes highly electrified. In concentrate H2SO4it is almost colorless, the presence of N oxides produces a dark green shade. In alc. KOH the color is dark red. Picrylaniline and Ag2O form a similar compound; reddish brown plates with a metallic luster from xylene, m. 278-80°. Alc. KOH, when added gradually to picryldiethylamine and pierYldibenzylamine, gives at first a dark red color which slowly becomes lighter as the concentrate of the alkali increases. Picryldibenzylamine, (O2N)3C6H3N(CH2Ph)2, from picryl chloride and dibenzylamine; slender, yellow needles from alc. or C6H6, in 173°. The following compds. were prepared from 2,4-dinitrodiphenylamine, PhNHC6H3(NO2)2: KOMe (see I); black needles with an intense violet luster. With EtOH + KOH a red amorphous substance is produced. With PrOH + KOH, aggregates of opaque, dark brown, highly unstable needles. Potassium isobutory derivative (see I), black, microscopic needles with a metallic luster. p-Nitrodiphenylamine is known to give a red color with alc. KOH, but excess of alkali does not cause the color to become lighter and the same is true of 2,4-dinitrodiphenylamine. The following compds. failed to react with alc. KOH: 2,4-dinitrodiphenylmethylamine (O2N)2C6H3NPhMe; 2,4-dinitrodiphenylethylamine and 2,4-dinitrophenyldimethylamine, but this latter compd., when warmed with C6H6and alc. KOH, is hydrolyzed to K 2,4-dinitrophenolate. 2,4-Dinitrophenylmethylamine, with C6H6and alc. KOH, gives an unstable, amorphous, dark red, pulverulent salt. "Trinitrobenzene" and also "trinitrotoluene" give red colors with alc. KOH, the colors become less intense with increasing alkali conc. and finally change to brownish or reddish yellow. Sym-Trinitrobenzene gives, with KOH and PrOH, the salt C15H2402N3K3; finely divided, red, unstable powder. A similar compound is obtained from 2,4,6-trinitrotoluene, red, amorphous and highly explosive. All the nitrolates are decomposed at once by H2O and also, but more slowly, on exposure to the atmosphere.
ACCESSION NUMBER: 1910:14710 CAPLUS
DOCUMENT NUMBER: 4:14710
ORIGINAL REFERENCE NO.: 4:2641i,2642a-i,2643a-e
TITLE: Salts of Aromatic Polynitro Compounds
AUTHOR(S): Busch, M.; Kogel, Walter
CORPORATE SOURCE: Chem. Lab.; Univ. Erlangen
SOURCE: Ber. (1910), 43, 1549-64
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

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AB see J. Chemical Society, 79, 522 (1901); 83, 1334 (1903); 89, 583 (1906). A study of a large number of addition products has resulted in the following conclusions: Primary arylamines in which the NH2 group is directly attached to the nucleus form colored additive compds. The depth of color is increased by the introduction of alkyl groups, especially in the p-position. The introduction of negative substituents does not necessarily inhibit the formation of an additive compound, but the colors are somewhat lighter. Primary arylamines of the naphthalene group form much more stable compds. than those of the benzene series. The presence of 2 or more NH2 groups in the arylamine mol. tend to deepen the color of the additive compds. The effect of introducing alkyl or aryl radicles into the NH2 group is noticeable. On the naphthalene and benzene series the tendency is for the introduction of aryl-alkyl groups to increase the depth of color. Tertiary amines from additive compds. provided not more than one aryl group is attached to the N-atomic When 2 groups are attached stable additive compds. cannot always be obtained. Quinoline and xyloquinoline form colored compds. Isoquinoline, o- and p-toluoquinoline and α- and β-naphthaquinoline form colorless or pale colored compds. Aniline and its homologues form well-defined compds. Aromatic amines, in which the NH2 group is attached to the side chain, and alkyl-arylamines generally give no compds. but all yield intensely red-colored liquids. The generalizations drawn by Kauffmann (Bie Auxochrome, Samm. chemical tech. Vortrage, XII, 2 (1907)) hold for these compds. The compds. made were: Trinitrobenzene with: o-chloroaniline, H2NC6H4Cl, C6H3(NO2)3, flat, orange prisms, m. 134.5°; m-chloroaniline, orange needles, m. 114.5°; p-chloroaniline, red prismatic needles, m. 110-1°; 2,4-dichloroaniline, bright red needles, m. 91°; s-trichloroaniline, yellowish brown needles, m. 93-4°; o-bromoaniline, orange-red needles, m. 128°; m-bromoaniline, orange-red needles, m. 115.5-6.5°; p-bromoaniline, scarlet needles, m. 113-3.5°; 2,4-dibromoaniline, orange needles, m. 86-6.5°; 2,6-dibromoaniline, canary-yellow needles, m. 104°; 2,4,6-tribromoaniline, orange-yellow needles, m. 111°; 2,3,4,6-tetrabromoaniline, yellow needles, m. 107.5-8°; α-bromo-β-naphthylamine, scarlet needles, m. 192-2.5°; acetyl derivative, yellow needles, m. 125°; 4-bromo-1-naphthylamine, brick-red needles, m. 195.5-6°; 1,6-dibromo-2-naphthylamine, m. 165°; o-nitroaniline, brownish-yellow needles, m. 91°; m-nitroaniline, yellow needles, m. 98°; α-nitro-β-naphthylamine, golden yellow needles, m. 115.5-6°; o-anisidine, brownish red plates, m. 98°; p-anisidine, black prisms or plates, m. 81-2°; Me o-aminobenzoate, orange-yellow needles, m. 106°; Et o-arninobenzoate, bright red needles, m. 71-1.5°; o-aminobenzoic acid, orange-yellow needles, m. 192-3°; K o-aminobenzoate, red needles, m. 114°; m-aminobenzoic acid, no compound, K salt, reddish brown needles, m. 118-9°; Et ester, m. 84-5°; Me p-aminobenzoate, orange-needles, m. 114-4.5°; Et derivative, scarlet needles, m. 85°; p-aminobenzoic acid, red crystalline, m. 151°; K salt, dark red needles, decompose 111°; p-aminoacetophenone, scarlet, flat prisms, m. 137.3°; ar-tetra-hydro-α-naphthylamine, brick-red needles, m. 113°; triaminotoluene, greenish black needles, decompose 159.5°; 2,4-diaminoazobenzene, black prisms, m. 144°; 3,3-diaminoazobenzene, reddish brown needles, m. 188°; p-aminobenzeneazodimethylaniline, brown plates, m. 157-8°; α-amino-β-naphthyl ethyl ether, purple black needles, m. 148°; 1-benzeneazo-2-naphthylamine dark red needles, m. 153°; 1,2-naphthylenediamine, purple needles, m. 203-4°; 1,4-naphthylenediamine, black needles, m. 208°, decomp.; the isomeric 1,5-diamine, brown needles, m. 245°; the 1,8-diamine, dark brown needles, m. 225°; Et 2-aminoindene-3-carboxylate, orange-red plates, m. 132.5°. Additive compds. of trinitrobenzene with secondary amines derived from C6H6 and naphthalene benzylaniline, red, hexagonal plates, m. 92°; benzyl-α-naphthylamine, chocolate-red needles, m. 174-4.5°; benzyl-p-naphthylamine, reddish-brown needles, m. 141° (with trinitrotoluene the above given crimson needles, m. 106.5°); phenyl-α-naphthylamine, purple needles, m. 130°; Ph-β-naphthylamine, reddish brown plates, m. 115.5°, contains 2 C6H3(NO2)3; another compd. forms with 1 mol. C6H3(NO3)3, brick-red needles, m. 109°; acetyl derivative, olive-green needles, m. 96-7° (Ph-α-naphthylamine and trinitrotoluene give dark red needles, m. 73-4°); α,α-dinaphthylamine, brown, prismatic needles, m. 156-7°; β,β-dinaphthylamine, brown prisms, m. 174°; o-tolyl-β-naphthylamine, crimson-red plates, m. 120.5-1°; p-tolyl-α-naphthylamine, dark brown plates, m. 72°, p-tolyl-β-naphthylamine, brick-red plates, m. 111-111.5°; Et β-anilinocrotonate, scarlet, flat prisms, m. 126°; β-imino-α-cyanohydrindene, black plates, m. 168-9°; formo-α-naphthalide, yellow needles, m. 160°; isomeric β-compd., yellow needles, m. 123°. Additive compds. with tertiary amines derived from C6H6 and naphthalene: dibenzyl-β-naphthylamine, purple black needles, m. 126-6.5°; corresponding compd. with trinitrotoluene, brick-red needles, m. 108°; dimethyl-p-aminobenzaldehyde, purplish brown needles, m. 91°; diethylamlnobenzylidene-p-aminomethylaniline, black plates, m. 162.5°; additive compds. with amines derived from di- and tri-phenylmethane, dibenzyl, etc.: o,o-diaminostilbene, purple brown needles, m. 190-1°; tetramethyl-p-diaminodiphenylmethane, black needles, m. 114-4.5°; tetramethyldiaminobenzhydrol, black needles, m. 75.5°; tetramethyldiaminobenzophenone, black needles, green metallic luster, m. 184-5°; tetramethyldiaminotriphenylmethane, black plates, m. 88.5-9°; tetramethyl-p-phenylenediamine, black needles, m. 142°. Additive compds. with Schiff's bases: benzylideneaniline, yellow, hexagonal plates, m. 112°; benzylidine-α-naphthylamine, brownish-yellow needles, m. 104°; benzylidine-β-naphthylamine, yellow needles, m. 150.5-151°. Additive compds. with phenylhydrazones: benzaldehydephenylhydrazone, red needles, m. 134°; m-nitrobenzaldehydephenylhydrazone, brick-red needles, m. 134.5°; cinnamaldehydephenylhydrazone, brownish red plates, m. 164.5°; acetonephenylhydrazone, reddish brown needles, m. 86.5-7°; acetophenonephenylhydrazone, dark red needles, m. 113.5°. Additive compds. with cyclic amines containing an N at. as part of the ring. α,α-diphenylpyridine, C23H16O6N4, lemon-yellow needles, m. 113°; γ,γ-dipyridyl, C5NH4C5NH4C6H3(NO2)3, is shown to exist by m. p. curves for mixtures of the 2 compds.; quinoline, colorless solid, m. 75°; isoquinoline, colorless, m. 87-8° (the last 2 compds. with o- and p-toluquinolines and quinaldine are formed by heating these compds. with trinitrobenzene. They are decomposed by solvents). α-Naphthaquinoline, yellow needles, m. 133.5°; isomeric β-compd. pale buff needles, m. 112°; tetrahydroquinoline, black plates, m. 100°; o-amino-p-toluquinoline, brick-red needles, m. 139°; carbazole, orange needles, m. 199.5°; (α-methylindole, yellow, m. 107°; acridine, amber needles, m. 115°; 3-phenylpyrazolone, canary-yellow prisms, m. 198°; 1-Ph-3-methylpyrazolone, ruby-red prisms, m. 92°. The additive

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compds. in general cryst. well and in many cases are decompd. by acids. They can be used for detection of small quantities of various amines and should prove of use in purification of many amines.

ACCESSION NUMBER: 1910:11773 CAPLUS
DOCUMENT NUMBER: 4:11773
ORIGINAL REFERENCE NO.: 4:2116d-i,2117a-i,2118a-b
TITLE: Additive Compounds of s-Trinitrobenzene with Arylamines. Combination as Affected by the Constitution of the Arylamine
AUTHOR(S): Sudborough, J. J.; Beard, S. H.
SOURCE: Journal of the Chemical Society, Abstracts (1910), 97, 773-98
CODEN: JCSAAZ; ISSN: 0590-9791
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L17 ANSWER 44 OF 49 CAPLUS COPYRIGHT 2005 ACS on STN
AB see J. Chemical Society, 79, 522 (1901); 83, 1334 (1903); 89, 583 (1906). A study of a large number of addition products has resulted in the following conclusions: Primary arylamines in which the NH2 group is directly attached to the nucleus form colored additive compds. The depth of color is increased by the introduction of alkyl groups, especially in the p-position. The introduction of negative substituents does not necessarily inhibit the formation of an additive compound, but the colors are somewhat lighter. Primary arylamines of the naphthalene group form much more stable compds. than those of the benzene series. The presence of 2 or more NH2 groups in the arylamine mol. tend to deepen the color of the additive compds. The effect of introducing alkyl or aryl radicles into the NH2 group is noticeable. On the naphthalene and benzene series the tendency is for the introduction of aryl-alkyl groups to increase the depth of color. Tertiary amines from additive compds. provided not more than one aryl group is attached to the N-atomic When 2 groups are attached stable additive compds. cannot always be obtained. Quinoline and xyloquinoline form colored compds. Isoquinoline, o- and p-toluoquinoline and α- and β-naphthaquinoline form colorless or pale colored compds. Aniline and its homologues form well-defined compds. Aromatic amines, in which the NH2 group is attached to the side chain, and alkyl-arylamines generally give no compds. but all yield intensely red-colored liquids. The generalizations drawn by Kauffmann (Die Auxochrome, Samm. chemical tech. Vortrage, XII, 2 (1907)) hold for these compds. The compds. made were: Trinitrobenzene with: o-chloroaniline, H2NC6H4Cl, C6H3(NO2)3, flat, orange prisms, m. 134.5°; m-chloroaniline, orange needles, m. 114.5°; p-chloroaniline, red prismatic needles, m. 110-1°; 2,4-dichloroaniline, bright red needles, m. 91°; s-trichloroaniline, yellowish brown needles, m. 93-4°; o-bromoaniline, orange-red needles, m. 128°; m-bromoaniline, orange-red needles, m. 115.5-6.5°; p-bromoaniline, scarlet needles, m. 113-3.5°; 2,4-dibromoaniline, orange needles, m. 86-6.5°; 2,6-dibromoaniline, canary-yellow needles, m. 104°; 2,4,6-tribromoaniline, orange-yellow needles, m. 111°; 2,3,4,6-tetrabromoaniline, yellow needles, m. 107.5-8°; α-bromo-β-naphthylamine, scarlet needles, m. 192-2.5°; acetyl derivative, yellow needles, m. 125°; 4-bromo-1-naphthylamine, brick-red needles, m. 195.5-6°; 1,6-dibromo-2-naphthylamine, m. 165°; o-nitroaniline, brownish-yellow needles, m. 91°; m-nitroaniline, yellow needles, m. 98°; α-nitro-β-naphthylamine, golden yellow needles, m. 115.5-6°; o-anisidine, brownish red plates, m. 98°; p-anisidine, black prisms or plates, m. 81-2°; Me o-aminobenzoate, orange-yellow needles, m. 106°; Et o-arninobenzoate, bright red needles, m. 71-1.5°; o-aminobenzoic acid, orange-yellow needles, m. 192-3°; K o-aminobenzoate, red needles, m. 114°; m-aminobenzoic acid, no compound, K salt, reddish brown needles, m. 118-9°; Et ester, m. 84-5°; Me p-aminobenzoate, orange-needles, m. 114-4.5°; Et derivative, scarlet needles, m. 85°; p-aminobenzoic acid, red crystalline, m. 151°; K salt, dark red needles, decompose 111°; p-aminoacetophenone, scarlet, flat prisms, m. 137.3°; ar-tetra-hydro-α-naphthylamine, brick-red needles, m. 113°; triaminotoluene, greenish black needles, decompose 159.5°; 2,4-diaminoazobenzene, black prisms, m. 144°; 3,3-diaminoazobenzene, reddish brown needles, m. 188°; p-aminobenzeneazodimethylaniline, brown plates, m. 157-8°; α-amino-β-naphthyl ethyl ether, purple black needles, m. 148°; I-benzeneazo-2-naphthylamine dark red needles, m. 153°; 1,2-naphthylenediamine, purple needles, m. 203-4°;

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1,4-naphthylenediamine, black needles, m. 208°, decomp.; the isomeric 1,5-diamine, brown needles, m. 245°; the 1,8-diamine, dark brown needles, m. 225°; Et 2-aminoindene-3-carboxylate, orange-red plates, m. 132.5°. Additive compds. of trinitrobenzene with secondary amines derived from C6H6 and naphthalene benzylaniline, red, hexagonal plates, m. 92°; benzyl-α-naphthylamine, chocolate-red needles, m. 174-4.5°; benzyl-p-naphthylamine, reddish-brown needles, m. 141° (with trinitrotoluene the above given crimson needles, m. 106.5°); phenyl-α-naphthylamine, purple needles, m. 130°; Ph-β-naphthylamine, reddish brown plates, m. 115.5°, contains 2 C6H3(NO2)3; another compd. forms with 1 mol. C6H3(NO3)3, brick-red needles, m. 109°; acetyl derivative, olive-green needles, m. 96-7° (Ph-α-naphthylamine and trinitrotoluene give dark red needles, m. 73-4°); α,α-dinaphthylamine, brown, prismatic needles, m. 156-7°; β,β-dinaphthylamine, brown prisms, m. 174°; o-tolyl-β-naphthylamine, crimson-red plates, m. 120.5-1°; p-tolyl-α-naphthylamine, dark brown plates, m. 724°, p-tolyl-β-naphthylamine, brick-red plates, m. 111-111.5°; Et β-anilinocrotonate, scarlet, flat prisms, m. 126°; β-imino-α-cyanohydrindene, black plates, m. 168-9°; formo-α-naphthalide, yellow needles, m. 160°; isomeric β-compd., yellow needles, m. 123°. Additive compds. with tertiary amines derived from C6H6 and naphthalene: dibenzyl-β-naphthylamine, purple black needles, m. 126-6.5°; corresponding compd. with trinitrotoluene, brick-red needles, m. 108°; dimethyl-p-aminobenzaldehyde, purplish brown needles, m. 91°; diethylamlnobenzylidene-p-aminomethylaniline, black plates, m. 162.5°; additive compds. with amines derived from di- and tri-phenylmethane, dibenzyl, etc.: o,o-diaminostilbene, purple brown needles, m. 190-1°; tetramethyl-p-diaminodiphenylmethane, black needles, m. 114-4.5°; tetramethyldiaminobenzhydrol, black needles, m. 75.5°; tetramethyldiaminobenzophenone, black needles, green metallic luster, m. 184-5°; tetramethyldiaminotriphenylmethane, black plates, m. 88.5-9°; tetramethyl-p-phenylenediamine, black needles, m. 142°. Additive compds. with Schiff's bases: benzylideneaniline, yellow, hexagonal plates, m. 112°; benzylidine-α-naphthylamine, brownish-yellow needles, m. 104°; benzylidine-β-naphthylamine, yellow needles, m. 150.5-151°. Additive compds. with phenylhydrazones: benzaldehydephenylhydrazone, red needles, m. 134°; m-nitrobenzaldehydephenylhydrazone, brick-red needles, m. 134.5°; cinnamaldehydephenylhydrazone, brownish red plates, m. 164.5°; acetonephenylhydrazone, reddish brown needles, m. 86.5-7°; acetophenonephenylhydrazone, dark red needles, m. 113.5°. Additive compds. with cyclic amines containing an N at. as part of the ring. α,α-diphenylpyridine, C23H16O6N4, lemon-yellow needles, m. 113°; γ,γ-dipyridyl, C5NH4C5NH4C6H3(NO2)3, is shown to exist by m. p. curves for mixtures of the 2 compds.; quinoline, colorless solid, m. 75°; isoquinoline, colorless, m. 87-8° (the last 2 compds. with o- and p-toluquinolines and quinaldine are formed by heating these compds. with trinitrobenzene. They are decomposed by solvents). α-Naphthaquinoline, yellow needles, m. 133.5°; isomeric β-compd. pale buff needles, m. 112°; tetrahydroquinoline, black plates, m. 100°; o-amino-p-toluquinoline, brick-red needles, m. 139°; carbazole, orange needles, m. 199.5°; (α-methylindole, yellow, m. 187°; acridine, amber needles, m. 115°; 3-phenylpyrazolone, canary-yellow prisms, m. 198°; 1-Ph-3-methylpyrazolone, ruby-red prisms, m. 92°. The additive

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compds. in general cryst. well and in many cases are decompd. by acids. They can be used for detection of small quantities of various amines and should prove of use in purification of many amines.

ACCESSION NUMBER: 1910:11772 CAPLUS
DOCUMENT NUMBER: 4:11772
ORIGINAL REFERENCE NO.: 4:2116d-i,2117a-i,2118a-b
TITLE: Additive Compounds of s-Trinitrobenzene with Arylamines. Combination as Affected by the Constitution of the Arylamine
AUTHOR(S): Sudborough, J. J.; Beard, S. H.
CORPORATE SOURCE: Univ. Coll., UK
SOURCE: Proc. Chem. Soc. (1910), 26, 71
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L17 ANSWER 45 OF 49 CAPLUS COPYRIGHT 2005 ACS on STN
GI For diagram(s), see printed CA Issue.
AB cf. preceding abstract p-Bromobenzoylacetic ester, prepared by Claisen's method from P-bromobenzoic ester, is a yellow oil. With HONH2 it gives p-bromophenyloxazolone; silvery lustrous plates from alc., decompose 118°. p-Bromophenylhydroximinoxazolone, formula (I) below, is formed from the preceding compound and HNO2; pale yellow crystals with 3 H2O from alc. + H2O. When anhydrous it is yellow and decompose 166°. In alc. the color is red, in other organic solvents yellow. The salts described below are quickly decompose by alkalies, more slowly by H2O, but are stable in alc. They were prepared from (I) and the base, or metallic alcoholate, in alc. Lithium salt, yellow. Sodium salt, orange-red; its solns. are deep violet. Monohydrate, light rose-colored. Potassium salts, rose-colored; bluish violet in acetone. Reddish violet needles or plates. Each salt gives a light red derivative with 1 PhOH. Acid salt, golden yellow. Rubidium salts, violet from alc. Blue from acetone. Rose-colored from acetone + CHCl2. Derivative with 1 PhOH, light red. Acid salt, golden yellow. Cesium salts, rose-colored, stable. Bluish, violet, labile. With 1 PhOH, light red. Barium salts, red with 4 H2O. Anhydrous, orange-colored. Calcium and magnesium salts, orange. Zinc salt, light yellow. Lead salt, light rose-colored. Thallium salt, flesh-colored. Silver salts, flesh-colored and unstable. Orange and crystalline. Blue salt, explodes with MeI. The resulting ether (II) is identical with that obtained from the orange-colored salt. A brown salt has also been obtained. Monohydrate, carmine-red. Dipyridine derivative, violet; when heated it evolves pyridine and becomes rose-colored. Diammonia compound, C9H4O3N2BrAg(NH3)2, deep blue. Monoammonia derivative, rose-colored. The compound with 1 MeCN is red, and changes to the above orange salt when warmed with alc. Mercurous and mercuric salts, yellow; orange-yellow in PhOH, orange-red in acetone and red in pyridine. Ammonium salt, orange needles. Methyl-, ethyl-, propyl- and benzylamine salts, rose-colored. Dimethyl- and diethylamine salts, red. Dipropylamine salt, orange. These last 3 salts are light red in CHCl3. Dibenzylamine salt, flesh-colored and labile. Red and stable. In CHCl3 the color is red, in pyridine violet. Trimethylamine salt, violet. Triethylamine salt, bluish violet. Both salts are reddish violet in CHCl2 or C6H6. Tripropylamine salt, red. Tetramethylammonium salt, deep bluish violet plates. Tetraethyl- and tetrapropylammonium salts, sky-blue plates. Pyridine and picoline salts, pale yellow. Methyl ether (II), from the Ag salts; pale yellow crystals from absolute Et2O + petroleum ether, m. and decompose 129°. Acetyl derivative, also from the Ag salts; yellow crystals from Et2O, m. and decompose 161°. Benzoyl derivative, decompose 167°. Anisoylacetic ester is prepared like the p-bromo compound described above, which it resembles. The reaction proceeds slowly and the yield is poor. Anisyloxazolone (III), from the ester; silvery lustrous crystals from alc., decompose 140-1°. Hydroxyiminoanisyloxazolone (IV), yellow needles with 1 H2O from H2O; when anhydrous decompose 149°. Sodium salt, orange-red; red in alc., violet in pyridine. Potassium salt, reddish violet needles. Caesium salt, bluish violet. Ammonium salt, cinnabar-red. Acid salt, yellow. Silver salts, labile, rose-colored; stable blue. Diammonia derivative, C10H7O4N2Ag(NH3)2, red. Methyl ether, light yellow, silky lustrous needles, m. 126°. When boiled with alkali it gives anisylfurazancarboxylic acid (V); crystals from H2O, m. 99-100°. The phenyl derivative is formed in a similar manner. A considerable number of the salts described above have had their mol. wts. determined in various solvents by different methods; in all cases the results agreed with the simple formulas. In nondissociating solvents the color deepens as the positive nature of the liquid, or of the metal or amine increases. The salts specially examined were (1) NH4, (2) Cs, (3) Rb, (4) K, (5) Na, (6) Li, (7) Ba+ 4H2O, (8) Ca, (9) Mg, (10) Zn. In PhOH, 1, 2, 3 and 4, red; 7, light red. In CHCl3, 1 violet. In acetone, and also in AcOEt, 1 and 2, blue; 3, violet-blue; 4, bluish violet; 5, violet; 6, carmine-red; 7, red. In pyridine, 1, 2 and 3, blue; 4, violet-blue; 5, bluish violet; 6, violet; 7 and 8, carmine-red; 9, orange-red; 10, orange-brown. Where no data are given the salts failed to dissolve. The absorption spectra of a number of the salts were detd. in various solvents and the results are reproduced in the form of curves. These indicate that the yellow salts of very feeble bases resemble the true hydroximinoketones in their structure, whereas the blue salts of the very strong bases are essentially similar to the nitrosoenolic type (cl. preceding and following abstrs.).
ACCESSION NUMBER: 1910:5242 CAPLUS
DOCUMENT NUMBER: 4:5242
ORIGINAL REFERENCE NO.: 4:923f-i,924a-i,925a
TITLE: Pantochromic Salts from Oximinooxazolones
AUTHOR(S): Hantzsch, A.; Heilbron, J.
SOURCE: Ber. (1910), 43, 68-82
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L17 ANSWER 46 OF 49 CAPLUS COPYRIGHT 2005 ACS on STN
AB The term pantochromic is applied to salts which occur in all colors and which are derived from colorless metals. When such a salt exists in 2 or more modifications, exhibiting different colors and varying degrees of stability, it is said to be chromotropic. The color of the solid salt may also be varied by the addition of solvent of crystallization and that of the solution by dissolving the salt in different "neutral" solvents. The salts described below have the general formula (I), where R = Me or Ph and M = a metal or ammonium group. Dimethylviolurates. Lithium salt: red from alc. It is deep red when anhydrous and red when it contains alc. or 1 H2O. Yellow salt from absolute MeOH. Yellow phenol derivative with 1 PhOH. Sodium salt. Red with 3 and 1 H2O. Anhydrous red and also violet. Red with 1 PhOH. Potassium salt, blue; violet with 0.5 H2O. Red with 1 PhOH. Rubidium salt, blue when anhydrous; bluish violet with 0.5 H2O; red with 1 PhOH. Cesium salt, indigo-blue needles without solvent of crystallization; red with 1 PhOH. Silver salt reddish brown; with the alkali salts it gives green and blue mixtures. With 1 pyridine a highly unstable green and also a stable bluish violet modification has been isolated. Methylamine salt, rose-colored. Acid salt, yellow. Dimethylamine salt, violet; in CHCl3 it is red. Trimethylamine salt, blue. Acid salt, orange-yellow. Tetramethylammonium salt, blue. Ethylamine salt, rose-red. Acid salt, yellow. Diethylamine salt, bluish violet; red in CHCl3, blue in pyridine. Triethylamine salt, bluish violet and unstable. Acid salt, orange-yellow. Tetraethylammonium salt, violet, becomes blue after solution in CHCl3, but regenerates the violet color on exposure to air. Propylamine salt, rose-red; red in CHCl3, blue in pyridine. Dipropylamine salt, bluish violet. Acid tripropylamine salt, N(C3H7)32C6H7O4N3, orange-yellow; violet in CHCl3, blue in pyridine. Tetrapropylammonium salt, greenish blue. Benzylamine salt, rosecolored. Dibenzylamine salt, labile form red; stable modification bluish violet. Piperidine salt, exists in 2 similar modifications. Pyridine salt, yellow. Acid salt also yellow. Dimethylvioluric acid is colorless but forms a yellow additive compound with 1 PhOH. Salts of diphenylvioluric acid. Lithium salt, red with 1 alc. and also when free from solvent. Yellow from MeOH. Sodium salt, carmine-red needles with alc.; reddish violet without solvent of crystallization Potassium salt, bluish violet with 1 alc.; reddish violet with.3 H2O; blue when anhydrous. Rubidium salt, indigo-blue needles with 1 alc.; reddish-violet with 3 H2O; blue when anhydrous. Acid salt, green. Cesium salt, blue crystals with a violet tinge containing 1 alc.; violet with H2O; blue when anhydrous. Acid salt, light green. Ammonium salt, deep violet needles with alc.; with H2O a reddish violet modification is produced. Silver salt almost colorless (leuco) labile salt, in H2O or alc. the color is violet; in acetone or CHCl3 red, pale greenish when dilute; in MeCN or pyridine, blue to bluish green. A violet highly labile salt was obtained once. The stable salt is dark green. Acid salt, orange crystals with 3 H2O. With pyridine green and blue modifications are produced. Thallium salt, unstable colorless form and stable, dark green modification. Magnesium salt, intensely yellow, red in pyridine. Zinc salt, yellow. Methyl diphenylviolurate, unstable, colorless and flocculent. The above results show that, in general, the color of the salts of the alkali metals passes from yellow through red and violet to blue, as the atomic weight of the metal increases. A similar change occurs in the case of the amine salts as the strength of the base increases. The influence of the solvent is marked; the color is changed towards the yellow with a negative solvent (PhOH), whereas a positive one ((pyridine) tends to impart a bluish-violet color. The mol. wt. of a number of the above salts of both acids was determined in various non-aqueous solvents, by the b. p. method; the results show that the compounds are monomol. The absorption spectra of many of the salts have also been determined in various solvents, the results being recorded in the form of curves. After a full discussion the conclusion is drawn that the blue salts are nitrosoenolic derivs. (II)
ACCESSION NUMBER: 1910:5241 CAPLUS
DOCUMENT NUMBER: 4:5241
ORIGINAL REFERENCE NO.: 4:922e-i,923a-f
TITLE: Pantochromic Dimethyl and Diphenylviolurates
AUTHOR(S): Hantzsch, A.; Robison, Robert
SOURCE: Ber. (1910), 43, 45-68
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L17 ANSWER 47 OF 49 CAPLUS COPYRIGHT 2005 ACS on STN
GI For diagram(s), see printed CA Issue.
AB A lengthy introduction gives the bibliography and a r.acte.esum.acte.e of the properties of N-amino heterocyclic compounds. When 2,3-naphthalene dihydrazine in alcohol was heated with 3 mols of p-isopropylbenzaldehyde, there was obtained di-p-ispropylbenzylidene-μ-p-isopropylphenyl-N-diamino-2,3-naphthodihydroglyoxaline, C40H42N4 (I), yellow needles from xylene, soluble in C6H6, C7H8, C5H10, insoluble in H2O, soluble in H2SO4 with a red color, m. 220°; boiled with HCl, NH4Cl and p-isopropylbenzaldazine were eliminated, yielding μ-p-ispropylphenyl-N-amino-2,3-naphthoglyoxaline, C20H20N3Cl (II), yellow-white needles from alcohol, colorless leaflets from AcOH, m. 249°, with decomposition; free base, C20H19N3, dirty white leaflets from alcohol, m. 265°, with decomposition; sulphate, C40H40O4N5S, light yellow needles, softens at 135°, does not m. 295°; nitrate, C20H20N4O3, yellowish white needles, m. 161°, with decomposition; picrate, C26H22N6O7, green-yellow needles, m. 223°; chlorplatinate, (C20H19N3)2.H2PtCl6, loam-yellow microscopic crystals, darkens at 240°, without melting; monacetyl derivative, C23H21N3O, colorless needles, m. 248°; picrylacetyl derivative, C28H24O8O6, needles, m. 270°; phenylthiosemicarbazide, C27H24N4S, prisms, m. 70°; benzylidine derivative, C27H29N3, yellow prisms, m. 15°; benzylidine hydrochloride, C27H24N3Cl, yellowish white needles, m. 244° with decomposition; benzylidine sulphate, C54H48N6SO4, needles, m. 150° with decomposition; nitrate, C27H24N4O3, yellow white needles, m. 160° with decomposition; picrate, C39H26N6O7, yellow needles, m. 228°; chlorplatinate, C54H48N6Cl6Pt, yellow crystals, m. 243° with decomposition; ethyliodide, C29H28N3I, yellowish red crystals, m. 179° with decomposition. With salicylic aldehyde, μ-p-isopropylphenyl-N-amino-2,3-naphthoglyoxaline yielded o-hydroxybenzylidine-μ-p-isopropylphenyl-N-amino-2-3-naphthoglyoxaline, C27H23N3O, yellowish white needles, m. 223°; with isopropylbenzaldehyde, p-isopropylbenzylidine-μ-p-isopropylphenyl-N-amino-2,3-naphthoglyoxaline, C30H29N3 (III) yellowish white needles, m. 260° with decomposition; with EtI, the ethyl iodide, C22H24N3I, yellow prisms, m. 199°. When benzidine-μ-p-isopropylphenyl-N-amino-2,3-naphthoglyoxaline was reduced with Zn + AcOH, it yielded **dibenzylamine** and μ-p-isopropylphenyl-2,3-naphthoglyoxaline, C20H18N2 (IV), white crystals, m. 247°; hyrochloride, C20H19N2Cl, yellowish white needles, m. 288° with decomposition; nitrate, C20H19N3O3, long yellowish white needles, m. 189° with decomposition; sulpate, C40H38O4N4S, white crystals, does not m. 295°; picrate, C26H21N5O7, yellow needles, m. 267°; chlorplatinate, C40H38N4Cl6Pt, yellowish red crystals.
ACCESSION NUMBER: 1908:10029 CAPLUS
DOCUMENT NUMBER: 2:10029
ORIGINAL REFERENCE NO.: 2:2250g-i,2251a-e
TITLE: N-Amino Heterocylic Compounds. (II) μ-p-Isopropyl-phenyl-N-Amino-2,3-Naphthoglyoxaline
AUTHOR(S): Franzen, Hartwig; Scheuermann, R.
SOURCE: Heidelberg. J. pr. Chem. (1908), 77, 193-225
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L17 ANSWER 48 OF 49 CAPLUS COPYRIGHT 2005 ACS on STN
GI For diagram(s), see printed CA Issue.
AB The authors have studied the condensation of camphoroxalic acid with secondary amines and have obtained compounds to which they assign the formula (I). Until the constitution is definitely settled, the authors suggest that these compounds be called isocamphoformolamine derivatives of the three types (II) camphoformeneamine, (III) camphoformolamine, and (IV) isocamphoformolamine, The isocamphoformolamine carboxylic acids all give a violet color with FeCl3 in alcohol solution and the acids or their salts when heated above their m. lose CO2 and water and yield camphoformeneamines which give no color with FeCl3. Diisobutylamine and camphoroxalic acid react at water bath temperature to form diisobutylisocamphoformolaminecarboxylic acid (see V) needles m. 179-80°. Heated above its m. it is converted into diisobutylcamphofomeneamine, (See VI) m. 73-4°. Diamylcamphoformolaminecarboxylic acid, C22H39O4N, crystals m. 160°. Diamylcamphoformeneamine, C21H37ON, plates, m. 43°. Diisoamylisocamphoformolaminecarboxylic acid, m. 156°. Diisoamylcamphoformenamine, m. 40°. **Dibenzylamine** and camphoroxalic acid at 130° give dibenzylcamphoformeneamine, C23H29ON, crystals m. 152°. Methylaniline and the acid react at 120° to form phenylmethylcamphoformeneamine, C18H23ON, tetrahedral crystals, m. 126°. Under like conditions ethylaniline yielded phenylethylcamphoformeneamine, C19H23OSN, oil, 110°, 285°. Benzylethylisocamphoformolaminecarboxylic acid, C21H29O4N, m. 158°. Benzylethylcamphoformeneamine C20H27ON, m. 57°. Acetylphenylhydrazine and camphoroxalic acid react at 140°' yielding acetylphenylaminecamphoformeneamine, (see VII) needles, m. 174°'. This is possibly the first acylated amine which has been induced to react with a ketonic or enolic compound. Negative results were obtained with camphoroxalic acid and a number of acyl derivatives of o-and p-aminophenol, as well as benzoylphenylhydrazine, benzylphenylhydrazine, benzylmethylamine, phenylmethylhydrazine, benzylaniline, phenyl-β-naphthylamine and p-phenylhydrazinesulphonic acid.
ACCESSION NUMBER: 1908:4274 CAPLUS
DOCUMENT NUMBER: 2:4274
ORIGINAL REFERENCE NO.: 2:1009f-i
TITLE: Study of the Action of Certain Secondary Amines on Camphoroxalic Acid (Eleventh Communication on Camphoroxalic Acid Derivatives)
AUTHOR(S): Tingle, J. Bishop; Williams, L. F.
CORPORATE SOURCE: McMaster Univ., Toronto
SOURCE: American Chemical Journal (1908), 39, 105-24
CODEN: ACJOAZ; ISSN: 0096-4085
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

L17 ANSWER 49 OF 49 CAPLUS COPYRIGHT 2005 ACS on STN
GI For diagram(s), see printed CA Issue.
AB The pyridine salt of hydroxymaleic anhydride, , m. 108°, with sulphuric acid of 12% yields hydroxymaleic acid. If the concentration of the sulphuric acid is 30%, hydroxyfumario acid is formed. **Dibenzylamine** hydroxyfumarate, C4H4O3.NH(C7H3)2, crystalline, m. and evolves carbon dioxide 127-128°. Hydrochloric acid, at the ordinary temperature, converts it into hydroxymaleic acid. Hydroxymaleic anhydride is an oil which could not be purified. Hydroxymaleanilic acid, PhNHCOCH: C(OH)CO2H, prepared at -15°; slightly yellow crystals, m and evolves gas 112-113°, gives a deep red color with ferric chloride. Sodium salt, granular crystals, soluble in 20 parts of water at 22°; m. and decomposes 156-158°. Hydroxyfumaranilic acid, prepared in a similar manner to the maleic derivative except that the crude aniline product is treated with 10 N sulphuric acid. Almost colorless crystals, m and decomposes 141-142°. It also gives a deep red color with ferric chloride. The reverse change of the fumaric into the maleic form is caused by treatment of the anilic acid with 5 N hydrochloric acid at -20°. Above -15° the addition of aniline to either of the anilic acids causes a more or less rapid evolution of carbon dioxide. (Cf. following abstract). Hydroxymaleicdibenzylaminic acid, (PhCH2)2NCOCH C(OH)CO2H, from the pyridine compound and **dibenzylamine**; colorless crystals m. and decomposes 147°.
ACCESSION NUMBER: 1907:10736 CAPLUS
DOCUMENT NUMBER: 1:10736
ORIGINAL REFERENCE NO.: 1:2558d-h
TITLE: Oxalacetic Acid
AUTHOR(S): Wohl, A.; Lips, C. H.
CORPORATE SOURCE: Org.-Chem. Lab. Tech. Hochschule, Danzig
SOURCE: Ber. (1907), 40, 2294-2300
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable

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